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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN No. 116.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS

OF THE

TWENTY-FOURTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

HELD AT

THE JAMESTOWN EXPOSITION, NORFOLK, VA.,
OCTOBER 9-11, 1907.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.



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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., May 18, 1908.

SIR: I have the honor to submit for your approval the Proceedings of the Twenty-fourth Annual Convention of the Association of Official Agricultural Chemists. All addresses and discussions have been briefly summarized and the reports submitted in the most concise form practicable. I recommend that these proceedings be published as Bulletin No. 116 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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PROCEEDINGS OF THE TWENTY-FOURTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

WEDNESDAY—MORNING SESSION.

The twenty-fourth annual convention of the Association of Official Agricultural Chemists was called to order by the president, Mr. J. P. Street, of New Haven, Conn., on the morning of October 9, in the assembly hall of the Inside Inn, Jamestown Exposition, Norfolk, Va.

The following members and visitors registered during the convention:

MEMBERS AND VISITORS PRESENT.

Allen, William M., Department of Agriculture, Raleigh, N. C.
Atkinson, Frederick C., District Chemist, Southern Cotton Oil Company, Savannah, Ga.
Averitt, S. D., Agricultural Experiment Station, Lexington, Ky.
Bailey, Herbert S., Bureau of Chemistry, Washington, D. C.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Birdsey, C. E., New York, N. Y.
Bizzell, James A., 804 E. Seneca street, Ithaca, N. Y.
Blair, A. W., Agricultural Experiment Station, Gainesville, Fla.
Browne, Charles A., Bureau of Chemistry, Washington, D. C.
Brinton, Clement S., U. S. Appraiser's Stores, Philadelphia, Pa.
Bryan, Thomas J., Illinois State Food Commission, Manhattan Building, Chicago, Ill.
Burnet, Wallace C., Bureau of Chemistry, Washington, D. C.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Carroll, John S., German Kali Works, Atlanta, Ga.
Cathcart, Charles S., Agricultural Experiment Station, New Brunswick, N. J.
Cook, Frank C., Bureau of Chemistry, Washington, D. C.
Cook, H. H., Waterbury, Conn.
Curry, B. E., Durham, N. C.
Davidson, Robert J., Agricultural Experiment Station, Blacksburg, Va.
Dodge, C. O., Washington, D. C.
Doyle, Aida M., 1025 Lamont street, Washington, D. C.
Dox, Arthur W., Assistant Dairyman, Bureau of Animal Industry, Storrs, Conn.
Edmonds, H. D., Storrs, Conn.
Ellett, Walter B., Agricultural Experiment Station, Blacksburg, Va.
Fraps, G. S., Agricultural Experiment Station, College Station, Tex.

- Frear, William, Agricultural Experiment Station, State College, Pa.
 Fuller, F. D., Department of Agriculture, Harrisburg, Pa.
 Fuller, Mrs. F. D., Harrisburg, Pa.
 Gaither, E. W., Nashville, Tenn.
 Gearkens, A., Antwerp, Belgium.
 Gudeman, Edward, Consulting Chemist, 704 Rialto Building, Chicago, Ill.
 Hand, William F., Agricultural Experiment Station, Agricultural College, Miss.
 Hanson, Herman H., Agricultural Experiment Station, Orono, Me.
 Hartwell, Burt L., Agricultural Experiment Station, Kingston, R. I.
 Haskins, H. D., Amherst, Mass.
 Haywood, William G., Department of Agriculture, Raleigh, N. C.
 Heide, Henry, 313 Hudson street, New York, N. Y.
 Herff, B. von, 93 Nassau street, New York, N. Y.
 Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
 Hopkins, Cyril G., University of Illinois, Urbana, Ill.
 Horne, W. D., National Sugar Refining Company, Yonkers, N. Y.
 Houghton, Harry W., Bureau of Chemistry, Washington, D. C.
 Howard, Burton J., Bureau of Chemistry, Washington, D. C.
 Jackson, H. C., Bender Hygienic Laboratory, Albany, N. Y.
 Kebler, Lyman F., Bureau of Chemistry, Washington, D. C.
 Kilgore, B. W., State Chemist, Raleigh, N. C.
 Lipman, Jacob G., Agricultural Experiment Station, New Brunswick, N. J.
 Lowenstein, Arthur, Morris & Co., Chicago, Ill.
 McDonnell, H. B., Maryland Agricultural College, College Park, Md.
 McGill, Anthony, Chemist to Canadian Government, Ottawa, Canada.
 Mason, Glen F., H. J. Heinz Company, Pittsburg, Pa.
 Michael, Louis G., Agricultural Experiment Station, Ames, Iowa.
 Moore, C. C., Department of Agriculture, Washington, D. C.
 Morrison, William G., F. S. Royster Guano Company, Norfolk, Va.
 Patrick, G. E., Bureau of Chemistry, Washington, D. C.
 Patten, Andrew J., Agricultural Experiment Station, Agricultural College, Mich.
 Patterson, H. J., Agricultural Experiment Station, College Park, Md.
 Pierce, Anne L., Bureau of Chemistry, Washington, D. C.
 Pinkerton, Thomas C., Walbrook, Baltimore, Md.
 Porch, Madison B., Hygienic Laboratory, Washington, D. C.
 Richardson, W. D., Swift & Co., Chicago, Ill.
 Richardson, Mrs. W. D., Chicago, Ill.
 Robb, John B., Department of Agriculture, Richmond, Va.
 Robertson, B. F., Agricultural Experiment Station, Clemson College, S. C.
 Rose, R. E., Tallahassee, Fla.
 Ross, B. B., State Chemist, Auburn, Ala.
 Rudnick, Paul, Armour & Co., Chicago, Ill.
 Schmitt, Walter, Armour & Co., Chicago, Ill.
 Shedd, O. M., Agricultural Experiment Station, Lexington, Ky.
 Sherwood, Sidney F., Bureau of Chemistry, Washington, D. C.
 Shutt, Frank T., Experiment Farm, Ottawa, Canada.
 Sindall, Harry E., 133 North Front street, Philadelphia, Pa.
 Smith, Frank M., H. J. Baker and Brother, 100 William street, New York, N. Y.
 Smither, F. W., Bureau of Chemistry, Washington, D. C.
 Snyder, Harry, Agricultural Experiment Station, St. Anthony Park, Minn.
 Street, John Phillips, Agricultural Experiment Station, New Haven, Conn.
 Taber, Walter C., Bureau of Soils, Washington, D. C.
 Tolman, L. M., Bureau of Chemistry, Washington, D. C.
 Trowbridge, P. F., Agricultural Experiment Station, Columbia, Mo.

Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
 Walker, Percy, Bureau of Chemistry, Washington, D. C.
 Warner, H. J., Department of Agriculture, Washington, D. C.
 Wiley, H. W., Bureau of Chemistry, Washington, D. C.
 Wiley, S. W., Wiley & Hoffman, Baltimore, Md.
 Willard, Julius F., Agricultural Experiment Station, Manhattan, Kans.
 Winton, A. L., Food and Drug Inspection Laboratory, Manhattan Building,
 Chicago, Ill.
 Withers, W. A., Agricultural Experiment Station, West Raleigh, N. C.
 Zerban, Fritz, Agricultural Experiment Station, Audubon Park, New Orleans, La.

A communication was presented from the Fertilizer Manufacturers' Association inviting the members of the Association of Official Agricultural Chemists to be their guests at dinner on the evening of October 9, and the secretary was instructed to accept the invitation on behalf of the association.

Letters from Atlantic City, N. J., Asbury Park, N. J., and Columbus, Ohio, inviting the association to hold the meeting of 1908 in the several localities named were also submitted and referred to the executive committee, the secretary being authorized to acknowledge the same.

FOOD ADULTERATION.

In the absence of Mr. Leach, referee on food adulteration, no general report on the subject was made, and the associate referees on the subject presented the subreports in the following order:

REPORT ON COLORS.

By E. F. LADD, *Associate Referee*.

The referee on colors submitted two subreports by Messrs. Hortvet and Gudeman and made the following recommendations, which were presented in his behalf by the secretary:

It is recommended—

(1) That more attention be given to the use of color standards in color work on foods by following the outline and method prepared by Mulliken in "A Method for Identification of Pure Organic Compounds," as suggested by Mr. Julius Hortvet.

(2) That a more comprehensive examination of the so-called "vegetable colors," some of which it would seem are not more properly to be classed as vegetable colors than are the anilin colors, be made during the coming year.

The paper submitted by Mr. Hortvet, entitled "An examination of colors used in foods or natural to foods," is to be found in the Eleventh Biennial Report of the Minnesota State Dairy and Food Commission, pages 583 to 593.

The second subreport, entitled "Solubilities and extraction values of food colors," by Edward Gudeman, is to be found in the Journal of the American Chemical Society, November, 1907, volume 29, No. 11, page 1629. The report contains two tables giving the solubility and extraction values obtained on three vegetable colors and three coal-tar colors of similar shades and possessing exceptional color intensities. The following extract is made, giving the conclusions reached:

Examination of these tables shows that the use of miscible solvents like methyl and ethyl alcohols and acetone are of no value for extraction purposes. Solvents of an acid-like character (ethyl acetate) are not suitable, as the results obtained are misleading, due to the effect of the solvent on the color.

The solubilities of many colors in petroleum ether, carbon disulphid, carbon tetrachlorid, and chloroform is so slight that these solvents are very suitable for preliminary extraction of food products, thereby extracting oils and fats before making dyeing tests to separate the colors. Care must be exercised, as many colors are soluble in fats or oils and are liable to be extracted in such preliminary treatment. Examination of the fats or oils will show whether any color has been extracted with these.

Conclusions drawn from a very large number of solubility and extraction tests, extending over a long period of time, are that the colors extracted or dissolved by many solvents under varying conditions from neutral, acid, or alkaline solutions, give no conclusive data for deciding upon the character or class of the colors themselves. The differences in solubility and in extractive values of vegetable colors, compared with coal-tar colors, are no greater nor less than the differences found between the various colors themselves belonging to the same class of colors.

Comparative color intensities were also determined, and it was found that only a very limited number of vegetable colors had a color intensity equal to one-fourth that of a corresponding shade of coal-tar color, and that the largest number of vegetable colors had one-tenth or less color intensity than the corresponding coal-tar color of similar shade.

In the absence of the referee on saccharine products, the following note from him was presented by the secretary:

REPORT ON SACCHARINE PRODUCTS.

By C. H. JONES, *Associate Referee.*

The referee on saccharine products attempted no cooperative work this year, but has personally examined many representative samples of maple sirup and sugar from the large crop of 1907 and found them to conform to the standards already established. It would seem that with the ash test, and the determination of the lead precipitate, and the malic acid value all ordinary maple substitutes might be readily differentiated from the pure article. A few changes in the determination of the malic acid value, based on the cooperative work done in 1906, have been referred to Mr. J. K. Haywood, chairman of the committee on the revision of methods, for insertion in the methods to be adopted at this convention.

REPORT ON COCOA AND COCOA PRODUCTS.

E. MONROE BAILEY, *Associate Referee*.

The associate referee on cocoa and cocoa products submitted the following methods of analysis by Winton, with the statement that with a few modifications they were the same as those employed at the Connecticut Agricultural Experiment Station, and published in the station report for 1902-3. He further suggested that the method used in the U. S. Department of Agriculture, Bureau of Chemistry (Dubois's method ^a), be substituted for section 13, "Sugar," and recommended that these methods be adopted by the association as provisional until the referee could make a fuller report upon this subject.

The methods, with the change in section 13 as suggested, are given in Bulletin 107, Revised, Methods of Analysis, page 254.

DISCUSSION ON SPICES.

The associate referee on spices, A. L. Winton, made no formal report, but stated that the methods for the examination of spices used at present seemed to him fairly satisfactory, and that the efforts of the association might therefore be better devoted to subjects of more pressing need.

Mr. Winton further said:

It has been my privilege to examine numerous shipments of spices in the warehouses of importers, where I have had an opportunity to note not only the general character of the products, but also the extent to which they have been damaged by insects. The nutmegs, known in the trade as grinding nutmegs, have been visited before shipment by some insect which, curiously enough, devours almost completely the starchy portion of the nut, but avoids the resinous veins. As a consequence, the nuts are light in weight and may be easily crushed between the fingers. Such nutmegs grind more easily than the sound product and, owing to the removal of the starchy matter, may contain a higher percentage of volatile oil; they are, however, unfit for consumption and their sale is a direct violation of the food and drugs act, on the ground that they are in whole or in part a filthy, decomposed, or putrid vegetable substance.

Whole ginger during long storage is almost sure to be attacked by the drug-store beetle, which makes numerous burrows through the hands and finally converts the product almost entirely into a disgusting powder.

Cayenne pepper and paprika, both before and after grinding, suffer severely from the attacks of a moth, the larva of which bores through the material and finally is transformed into the adult stage with the formation of a web. On opening a bottle of cayenne pepper which has been closed for months or even years a number of these moths will often fly out of the bottle.

Mr. KEBLER. Considerable quantities of worm-eaten ginger are imported into the United States. Several samples were detained at the ports, and at the hearing it developed that the worm-eaten ginger was ground and used in the manu-

^a J. Amer. Chem. Soc., 1907, 29: 556; U. S. Dept. Agr., Bureau of Chemistry, Bul. 107, Rev., Appendix, page 256.

facture of ginger cookies and similar products. Most of the so-called Calcutta ginger is of inferior quality, being usually mixed with abnormal quantities of dirt, the ginger itself never having been cleaned.

A few days ago I visited a large drug warehouse and inspected a number of deliveries of ginger, and in no case was there any material amount worm-eaten. Some of the Jamaica ginger had been slightly attacked by insects, but a large lot of African ginger which had been in the warehouse over four years did not show the slightest indication of such deterioration.

Mr. MCGILL. Is oil of nutmeg an article of commerce? I have not happened to find it on the Canadian market. I can conceive that nutmegs might even be increased in value for purposes of oil extraction by the removal of nonstarch matters by worms or otherwise. There are probably in attendance some persons who can give first-hand information as regards this question.

Mr. KEBLER. Not only is a considerable amount of nutmeg oil itself used, but the oil obtained from the mace is also employed in the manufacture of certain commodities.

FLAVORING EXTRACTS.

No report was submitted by the associate referee on flavoring extracts. The following recommendation in regard to the work for next year on this subject was offered by C. S. Brinton:

In view of the requirements of the new food and drugs act, and the standards adopted by the Department of Agriculture, it is necessary that we should have more definite information on this subject, especially on vanilla extracts. The standards require the soluble matter of 10 grams of vanilla beans in each 100 cc of finished extract. I therefore recommend that the associate referee on this subject for next year be instructed to investigate, and, if possible, to devise new methods for the detection of caramel, and also to investigate the composition and quantity of the soluble matters obtained from vanilla beans in preparing vanilla extract.

The associate referee on cereal products, A. McGill, of Ottawa, Canada, reported that the research work he had undertaken early in the year was not yet completed, because of the new duties which he had assumed upon the death of the chief analyst of Canada, the direction of the laboratories necessitating that the investigations referred to be laid aside.

REPORT ON PRESERVATIVES.

By W. D. BIGELOW, *Associate Referee.*

SALICYLIC ACID.

During the last year a question has arisen as to the applicability of the method commonly used for the detection of salicylic acid in dark colored malt liquors and other products colored dark brown by heat. It has been claimed by manufacturers that errors have occurred because of the similarity of the color reaction with ferric chlorid and with salicylic acid. Under the circumstances it seemed advisable to investigate carefully the application of the methods for the detection of salicylic acid to such products.

Samples were obtained of beer and other malt liquors manufactured from highly colored malt and of breakfast foods colored by heating to a high tem

perature. These samples were examined independently by A. M. Doyle and P. B. Dunbar of the Bureau of Chemistry, neither of whom had any difficulty in detecting added salicylic acid in any of the products.

It was found that on the addition of a ferric chlorid solution directly to the residue in the dish in which the ether extract was evaporated a dark red or purplish color was sometimes formed. If the dried ether residue was first dissolved in water, however, and the ferric chlorid added to the solution, no difficulty was experienced in any case. Very small amounts of salicylic acid were sometimes obscured by the color of the solution, but no color was obtained in the absence of salicylic acid which would lead the analyst to suspect its presence. A positive reaction for salicylic acid may be confirmed by the Millon reagent, which gives a light pink reaction in the absence of salicylic acid and a deep red in its presence. The ferric chlorid reaction was regarded by both observers as superior in such products to that of the Millon reagent.

FORMALDEHYDE.

The influence of the strength of the hydrochloric acid employed in the hydrochloric acid—ferric chlorid reagent suggested by Leach has been studied during the last year by A. V. H. Mory. It is known that the purple color given by proteids when heated with strong hydrochloric acid is a disturbing factor in the detection of formaldehyde in milk, and various methods have been adopted to avoid error or uncertainty. Some analysts have been accustomed to dilute the hydrochloric acid considerably, others to maintain a temperature sufficiently low to prevent the formation of a proteid reaction, and still others to guard against the difficulty by limiting the time of heating. In order to determine whether such dilution of the acid as would make the proportion of acid to milk a negligible quantity could be made without seriously interfering with the delicacy of the test for formaldehyde, the following dilutions of acid, each containing the specified amount of ferric chlorid, were heated to the boiling point with formaldehyde-free milk, in the proportion of two parts of the acid reagent to one of milk. This temperature and ratio were used because it is very easy to overheat, and the quantities used are generally not measured; also because excess of heat and of acid are known to be the chief sources of trouble.

Hydrochloric acid (sp. gr. 1.19).	Water.
cc.	cc.
100	-----
90	10
80	20
70	30
60	40

These mixtures were heated with stirring over a free flame in a casserole. The strongest acid gave a deep purple color, which began to develop below 70° C., and deepened quickly to a dark purple. The weakest acid gave practically no color even on boiling and after standing, while the intermediate strengths gave colors increasing regularly in depth and in purple tint with increase in acid strength.

The same experiment, except that milk containing a small quantity of formaldehyde (1 in 250,000) was used, showed a marked falling off in the delicacy of the test with dilution of the acid.

The general results of other experiments along this line show that if equal (measured) quantities of strong acid (sp. gr. 1.19) and milk be used and the

heating not carried beyond 90° C., little or no color is given by formaldehyde-free milk; but if, on the other hand, the acid is in excess or the heating is carried too near the boiling point, the proteid reaction becomes a factor increasing with the excess of acid and with the temperature.

Applying these results to the method referred to, it would appear to be desirable to replace the expression "about" [line 1 (e), p. 185, Bul. 107, Rev.] with the specification that equal (measured) quantities of the strong acid reagent and milk must be used, and to warn the analyst against the excessive use of either acid or heat, since either may produce a color hardly distinguishable from that given when formaldehyde is present.

The fact that the color given by formaldehyde develops much more quickly than that given by proteids alone may also be taken as a rough guide, but is of little value when the acid is in great excess.

SULPHUROUS ACID.

A great deal of attention has been given during the last year by the Division of Foods of the Bureau of Chemistry and the branch laboratories in San Francisco and New Orleans to the determination of sulphurous acid in dried fruit and molasses. In connection with the work, various methods for the determination of sulphurous acid were carefully studied in all three laboratories. In the New Orleans laboratory the work was done by A. Hugh Bryan with the assistance of C. O. Dodge and C. M. Boyles; in the San Francisco laboratory the work was done by Ralph A. Gould, E. J. Lea, and others; in the Bureau of Chemistry at Washington by M. C. Albrech, F. W. Liepsner, P. B. Dunbar, and C. P. Wilson.

It was found that the most satisfactory results were obtained with a long-necked, round-bottomed Kjeldahl flask of 500 cc capacity. Twenty grams was the maximum amount of dried fruit which could be used advantageously. On the addition of water the fruit swelled up so that a larger amount caused difficulty in boiling. In the case of molasses, 50 grams was the maximum amount that could be advantageously distilled. The use of larger samples in correspondingly larger flasks was found to be unsatisfactory.

Foaming, which occurs in both classes of products, appears to be best controlled with paraffin. Sodium chlorid deters the foaming with dried fruit to a certain extent, but the benefit derived from its use does not appear to be uniform. A current of carbon dioxid when introduced above the liquid materially decreases foaming, but when it enters at the bottom of the flask foaming seems to be increased.

It has been suggested by C. S. Ash that the volumetric method be modified by distilling into a normal solution of potassium hydroxid instead of into a standard iodine solution as is customary. The experiments by Messrs. Gould and Lea do not indicate that this method is advantageous. The average of the results obtained by it are very slightly higher than those obtained by distilling into iodine, but such results are somewhat irregular and duplicates are not altogether satisfactory. If it is impossible to titrate immediately after distillation the potassium hydroxid method has an advantage, as the oxidation of the sulphite occurs very slowly. Some delay may, therefore, follow the distillation in such case. It is essential, however, that titration follow immediately the acidification of the distillate. In case of distilling into iodine, the distillate should be titrated immediately.

Objections to the volumetric method have been repeatedly made, especially by Horne,^a because of the hydrogen sulphid said to be eliminated from molasses

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 125.

on boiling with acid. It has been recommended that the hydrogen sulphid be eliminated by passing the distillate through a trap containing cadmium chlorid or copper sulphate. The results obtained by the use of the trap with both dried fruit and molasses were found to be unsatisfactory. The efficiency of the apparatus was often somewhat reduced, although such reduction did not appear to be due to the presence of hydrogen sulphid, as no deposit of sulphid was found. A slight black deposit was sometimes found in the case of the molasses samples, but examination showed that it did not consist of copper sulphid. It therefore appears to be definitely proven that the amount of the hydrogen sulphid eliminated in the determination of sulphurous acid in dried fruits and molasses is so slight as not to interfere with the accuracy of the operation.

Zerban (page 77) has found that satisfactory results in the determination of sulphur dioxid in molasses can only be obtained by diluting 50 or 100 grams of the sample to 1,000 cc and distilling 800 cc. Even then an additional amount of distillate would contain some sulphur. Bryan and his associates found it unnecessary to employ such dilution or to distil so great a volume. His method was to dilute 50 grams of molasses with 100 cc of water, distil until the foam turns darker, add by means of a separatory funnel 3 portions of water of 50 cc each, and continue the distillation as long as possible after each addition. It was found that practically complete results were obtained by this method.

In the case of dried fruit, Gould found that satisfactory results could be obtained by treating 16 grams of the sample with 175 cc of water and distilling to a small volume. From the consequent concentration more complete results are obtained than from the distillation of a much larger but less concentrated volume.

Practically complete results were obtained in Washington by treating 20 grams of dried fruit with 300 cc of water and distilling 150 cc. Almost all of the sulphur dioxid was contained in the first 100 cc of the distillate, and the amount contained in portions of the distillate following the first 150 cc contained sulphur dioxid in negligible quantity, if at all.

It was demonstrated in all three laboratories that the greatest care must be exercised to prevent charring, especially if the volumetric method is employed. Charring evidently produces a reducing substance which gives a false indication regarding the amount of sulphur dioxid present. Bryan suggests guarding against this by placing the flask upon an asbestos board and exposing but a small surface to the flame. In all three laboratories, and especially in San Francisco and New Orleans, a careful and exhaustive comparison was made of the reliability, efficiency, and accuracy of the volumetric with the gravimetric method. The results obtained with the gravimetric method are slightly higher in the presence of a small amount of sulphur dioxid. In the presence of larger amounts the results are comparative in both molasses and dried fruits. Comparative results with the two methods were obtained in New Orleans with molasses containing 380 milligrams and more of sulphur dioxid per kilogram.

Gould and Lea studied carefully the sources of inaccuracy in the volumetric method which depends on receiving the distillate in iodine and titrating the residual iodine. They found that the chief source of inaccuracy lay in the volatilization of the iodine which is considerable when a current of carbon dioxide is employed. This volatilization was most pronounced when a beaker was used as a receiver, but was excessive even when a flask was employed. Traps were ineffective when attached to an ordinary flask, as cork stoppers were found to absorb quite an amount of iodine and rubber stoppers were still more objectionable.

The best results by the volumetric method were obtained with 8-ounce Drechsel wash bottles, attached to a trap provided with 5 cc of N/50 thiosulphate

solution. By this means the loss was reduced to 0.2 cc of N/50 iodine solution. The efficiency of the gravimetric method with both peaches and molasses is much greater in the presence of a large amount of sulphur dioxide than in the presence of a small amount. According to the results obtained in Washington, in the presence of 200 milligrams of sulphur dioxide per kilogram of fruit, the efficiency amounts to but 45 per cent; with 500 milligrams, to 70 per cent; with 1,000 milligrams, to from 70 to 75 per cent, and with 2,000 milligrams to about 85 per cent of the sulphur dioxide present. This was as large a percentage as could be recovered regularly with dried fruit. In the New Orleans laboratory the efficiency ranged from 83 to 89 per cent in molasses whose content of sulphur dioxide varied from 100 to 3,300 milligrams per kilogram. As with the dried fruit, the greater efficiency was secured in the presence of the larger amount of sulphur dioxide.

According to the results obtained both at San Francisco and New Orleans, the use of carbon dioxide may be greatly reduced. It can be advantageously employed to remove the air from the flask before beginning the distillation, but the current may then be stopped without sacrifice. Mr. Bryan found that equally good results were obtained by dispensing with the current of carbon dioxide and using instead a solution of sodium bicarbonate, the acidity being increased correspondingly. The purity of the reagent should be tested by making blank determinations for sulphur trioxide, as some blanks will run as high as 50 to 60 mg per kilogram. In the New Orleans laboratory blanks were made daily and the results subtracted from the determinations for that particular day.

LEGISLATION ON PRESERVATIVES IN FOOD.

By H. L. HARRIS, *Pacific Coast Borax Company.*

When William H. Moody was Attorney-General he said: "A thing may be within the statute but not within the letter, or within the letter and not within the statute. The intent of the lawmaker is the law." The pure food law was enacted for the purpose of protecting consumers from being defrauded or injured by adulterated food products, and to compel merchants to truthfully label their goods. Among the matters it aims to regulate is the use of preservatives in food. By authority of this law Food Inspection Decision 76 says: "It has been determined that no drug, chemical, or harmful or deleterious dye or preservative may be used. Common salt, sugar, wood smoke, potable distilled liquors, vinegar, and condiments may be used." It is evident that the food inspectors consider salt, sugar, smoke, alcohol, vinegar, etc., innocent, wholesome preservatives. I do not suppose that they would have us believe that these substances may not, under some conditions, be injurious. In regard to this latter point various authorities express themselves as follows:

SALT.

The United States Dispensary, page 1249: "Sodium chlorid in small doses acts as a stomachic tonic and anthelmintic, in larger ones as a purgative and emetic."

Food and Dietetics, Hutchison, page 291: "It has been asserted, for instance, that the addition of salt to the food aids digestion (Ogata), but more recent and exact experiments have shown that in health, at least, and in moderate doses salt has very little real influence on digestion at all, while in large quantities it actually delays the process."

Practical Hygiene, Harrington, page 209: "In the process of salting, the soluble organic constituents of meat and fish are removed in large part, and the fibers become hardened. The nutritive value and digestibility therefore are diminished correspondingly. Brine salting of fish is one of the oldest processes of preservation known."

Farmers' Bulletin 183, page 29: "Salt is an astringent, and when applied alone to meat renders it very hard and dry. Its action is first to draw out the meat juices. In a few days it will contract and harden the muscle fibers, thus shrinking the volume of meat."

Salt is a natural constituent of the blood. In small quantities it is, without question, beneficial and even necessary to health. When it is used, however, in large quantities to cure or preserve meats, fish, etc., it undoubtedly lessens the flavor, diminishes the food value, and renders food thus preserved or cured more difficult to digest. Unless its use is properly controlled it may be distinctly injurious to health.

SUGAR.

Food and Dietetics, Hutchison, page 275: "In strong solution sugar is an irritant to the tissues. In contact with the skin it is apt to set up superficial inflammation. This is familiar in the form of eczema, which is apt to appear in diabetics from the contact of the sugar-containing urine with the skin, and from the similar condition occurring on the arms of grocers and other persons who have frequently to handle sugar, and it is on account of its irritating properties that sugar can not be used as a subcutaneous aliment. * * * The same is true of the stomach. Brandl, experimenting on dogs, found that a 5.7 per cent solution of sugar produced reddening of the mucous membrane; if the concentration was increased to 10 per cent the mucous membrane became dark red, while a 20 per cent solution produced pain and great distress. This irritating effect on the mucous membrane is accompanied by the production of much mucus and the pouring out of a highly acid gastric juice."

The above evidence clearly indicates that sugar may be injurious to the cuticle or mucous membrane.

SMOKE.

Practical Hygiene, Harrington, page 209: "Smoking consists in exposing the meat or fish to the action of the smoke of wood fires, after, as a rule, a preliminary salting. The exposed material, already deprived of part of its natural moisture, becomes dried still further, and is partly penetrated by acetic acid, creosote, and other preservative elements of smoke."

Preservatives in Food and Food Examination, Thresh and Porter, page 9: "Several articles of food are frequently preserved by being exposed to the smoke from smoldering wood or sawdust. During this process they are partially dried and the material absorbs certain antiseptic substances from the smoke. * * * Creosote is probably one of the active antiseptic agents. It is a very poisonous substance, and doubtless a great outcry would be raised were anyone to attempt to use it for preserving food, but so long as it is introduced into the food in an old-fashioned manner, no objections are raised. It is only when some one wishes to improve upon ancient methods that the effect of prejudice and conservatism makes itself felt. It has never been alleged, so far as we are aware, that smoked meat is unwholesome, though its digestibility is almost certainly impaired. Any modern system of preserving which affected the digestibility to a similar degree would be strongly condemned."

ALCOHOL.

Food and Dietetics, Hutchison, page 343: "The bad effects of alcohol taken in quantities sufficient to produce intoxication are too apparent to require to be insisted upon. It must be remembered, however, that the habitual consumption of alcohol in quantities which, though insufficient to produce any outward and visible signs of intoxication, are yet beyond the immediate oxidizing power of the cells, may end by playing havoc with the tissues. Here, again, the brain seems specially liable to suffer, probably owing to its being one of the most highly organized and delicate tissues of the body. * * * Throughout the body generally the presence of even a slight amount of undecomposed alcohol leads to a diminution of the chemical energy of the cells, which interferes with the ordinary course of metabolism, and may result in chronic disease."

VINEGAR.

Food and Dietetics, Hutchison, page 284: "Although the acetic acid which vinegar contains is ultimately oxidized in the body, with the production of alkaline compounds, there is still reason to believe that it has an unfavorable influence in gout, and may even precipitate an attack if freely indulged in."

Preservatives in Food and Food Examination, Thresh and Porter, page 93: "Vinegar is a very popular condiment and preservative. Taken in excessive quantities it interferes with the digestive processes, and its use in such quantities, if persisted in, ultimately causes emaciation. Used in moderation, however, it is not likely to produce injurious consequences; nevertheless, if it were not one of the oldest preservatives in use, objections would be raised to its introduction. The effect of excessive quantities on adults and children and its potential effect on invalids would be dilated upon, and its use probably condemned."

It follows from the above facts that the preservatives deemed innocent are, under certain conditions, capable of producing distinct injury to the digestive system. The use of certain other preservatives is not permissible under Food-Inspection Decision 76. Among these the boron preservatives are included. In the effort to eliminate harmful substances from food much has been said about the use of boron preservatives, and the conclusion has been reached by some that they are actually harmful. After a most careful, and, as far as possible, unprejudiced consideration of the facts in the case, and after advice with many professionally competent to form an opinion upon this question, it is my honest conviction that boron preservatives are less injurious to health than the substances permitted by Food-Inspection Decision 76. If this is true, the fact will in time be recognized. When it is recognized we will want to make use of the available means that are most efficient. The boron preservatives far outweigh, in their preserving properties, the substances permitted by Decision 76. True, if the question of quantity is ignored, and if conditions not existing in preserved foods are considered, facts may be arrayed against boron preservatives precisely as we have found them in the case of common salt, sugar, wood smoke, potable distilled liquors, and vinegar. But if such facts are not prohibitive against these preservatives of the ancients, why should they be given greater weight against our modern preservatives? While it is true reports have been circulated that boric acid and borax have been injurious to health, when such reports are scientifically investigated the facts invariably show that it was not food preserved with boron compounds, but that excessive doses had been administered medicinally. Boron compounds should not be condemned as food

preservatives on account of excessive quantities having been administered medicinally to invalids.

Let us look critically and honestly at the latest report of an assumed poisonous action of borax. In the *Lancet* of August 10, 1907, page 369, we read: "A fortnight after birth the child developed thrush, for which borax and honey were applied. The child seemed to be relieved of the thrush by this remedy and developed such a liking for it that it was applied most liberally, from two to three four-drachm boxes having been used every week from the second to the eighth week."

The article states that the child wasted away, and that there was no cause for the wasting and rash except the borax, of which the child had about 10 grains every day for six weeks. I do not wish to seem to advocate the administration of borax in this amount to an infant of two to eight weeks, but even with its use does the evidence establish the fact that it acted injuriously? The existing condition of thrush shows that the child was ill before the administration of borax. What was responsible for that? Was the child suffering from a catarrhal stomatitis, perhaps as a result of improper feeding, or was the thrush due to want of cleanliness of the mouth? In the latter event, is it not quite as just to maintain that the child wasted away because of a condition that might have been prevented by the proper use of a solution of borax for keeping the mouth clean? If so, it was the lack of borax before the disease, and not its use afterwards, that was the cause, so far as the borax is concerned; at least, there are two points of view in the matter. If injury was caused by the medicinal application containing borax, may not the honey have been at fault rather than the borax? In speaking of honey, Harrington, in *Practical Hygiene*, page 166, says: "Honey from the flowers of the yellow jasmine has been known to produce serious and even fatal results."

Diet in Health and Disease, Friedenwald and Ruhrah, page 188: "Honey may contain poison. Plugge found that the honey from the *Rhododendron ponticum* is poisonous, and Xenophon, in his *Anabasis*, describes attacks of intoxication due to eating honey. Although death seemed near, none of his soldiers were killed by it. Strabbo and Dioscorides both speak of honey as producing madness or melancholia. * * * The honey from gelsemium is also poisonous. In Branchville, S. C., twenty persons were made ill and three died from eating honey derived from this source. In New Zealand honey from the 'whauriki,' a cress-like plant, causes severe symptoms and sometimes death."

When the poisonous effects of honey are so well known, was it wise to feed a two-weeks-old infant comparatively all the borax and honey it could swallow, and was it right to conclude that borax was the agent that caused the illness? I believe that there is no more authentic evidence than this of boron-preserved foods ever having caused disease in persons because of the boron present. On the contrary, there is ample evidence that there is a vital necessity for the proper preservation of perishable articles of food.

According to the press reports there have been, during the past nine months, over 4,700 cases of ptomaine poisoning in the United States, 119 of which were fatal. There have doubtless been many more that were not reported by the press. All such cases were preventable by proper hygienic measures. Under the above circumstances, is it just to condemn modern methods of preserving food upon what, at most, is uncertain evidence?

I sincerely trust, gentlemen, you will carefully consider the above facts, and realize that food which poisoned thousands of our fellow beings is not pure food, and that it deteriorated and became impure and poisonous owing to the fact

that it was not preserved; and, further, that a judicious use of boron compounds will protect the lives and health of the public by keeping food in a sweet, healthful, hygienic condition. The above facts should appeal to you so forcibly that you will at once realize the value, necessity, and innocence of boron compounds when used for the preservation of food.

The following committees on recommendations of referees were appointed by the president:

Committee A.—R. J. Davidson; Harry Snyder; H. D. Haskins.

Committee B.—B. B. Ross; F. D. Fuller; Harry Snyder.

Committee C.—L. M. Tolman; A. L. Winton; A. McGill.

The convention adjourned to meet at 9.30 Thursday morning.

SECOND DAY.

THURSDAY—MORNING SESSION.

At the opening of the session the following papers were presented, which, together with the report of the associate referee on the determination of water in foods, completed the subject of food adulteration.

Mr. C. B. Cochran submitted an outline of the classification of coal-tar colors to serve as a basis for future investigations, with the idea that, if possible, a committee be appointed to assist in the prosecution of such an investigation. In this connection the following paper by Mr. Cochran is also submitted:

THE ACTION OF SODIUM BISULPHITE REAGENT ON CERTAIN COAL-TAR COLORS.

By C. B. COCHRAN.

The sodium bisulphite reagent is made by saturating a 5 per cent solution of sodium hydroxid with sulphur dioxide. It is therefore a solution of $\text{NaHSO}_3 + \text{H}_2\text{SO}_3$. This reagent added to a water solution of any of the following coal-tar colors, decolorizes the solution, and on heating the color reappears but disappears again on cooling. The heating and cooling may be repeated many times and each time the color will reappear in the solution while hot.

Acid magenta (A) S. and J. 462.

Fuchsin (A) S. and J. 448.

Methyl violet (Grübler) S. and J. 451.

Ethyl violet (Grübler) S. and J. 453.

Guinea violet 4B (A).

Ethyl green (A) S. and J. 428.

Guinea green B (A).

Chrome green S. and J. 443.

Acid green (Grübler) S. and J. 435.

Malachite green (Grübler).

Turquoise blue BB.

Acridin red (Grübler).

The references to Schultz and Julius are taken from the 1904 edition. (A) indicates the Berlin Aniline Company.

With the exception of acridin red, all the colors in this list belong to Group X of Schultz and Julius which comprises the triphenyl methane and diphenyl-naphthyl methane coloring matters. The list is taken from about two hundred colors examined and includes all those which were found to exhibit the peculiar behavior with sodium bisulphite reagent which has been briefly described.

The only color of Group X thus far examined which has shown a different behavior with sodium bisulphite reagent is China blue (Grübler) S. and J. 480. This color is readily bleached, but the color does not seem to return on heating.

The results of the experiments made with these colors proved that the temperature at which the decolorized solution began to show a return of the color depended on the amount of sodium bisulphite reagent used. It was also found that, in many cases at least, if this reagent was added in considerable excess of the amount required to bleach the solution, no color would return even on heating to the boiling point.

The following description of the results of some of the experiments made on two of the colors will illustrate in a general way the behavior of the above-named coal-tar colors when treated with this reagent:

Acid magenta (A).—A solution of this color was made of such a depth that printed letters one-eighth of an inch long could just be distinguished in strong daylight through a layer of 5 inches.

Experiment I: To 5 cc of this solution was added 1 cc of sodium bisulphite reagent. The solution was decolorized within a few seconds. On heating to the boiling point only a faint color returned, which quickly disappeared on cooling.

Experiment II: To 5 cc of solution 0.5 cc of the reagent was added. The color disappeared in two or three minutes. On heating to the boiling point a deep color returned, which disappeared at 50° C.

Acridin red (Grübler).—The solution used was made of the same depth as in the case of acid magenta.

Experiment I: To 5 cc of solution 1 cc of reagent was added. The color disappeared at once. Only a faint color returned on heating to the boiling point.

Experiment II: To 10 cc of the solution 0.1 cc reagent was added. The color disappeared in one minute, but returned in its original intensity on heating to the boiling point and did not entirely disappear until the solution was cooled to 27° C.

Ethyl green (A).—This color is very easily bleached, and if a great excess of the reagent is used no color returns even on heating to the boiling point and continuing the heat for some time. When only a slight excess of the reagent is present a strong green color returns on heating to 60° C., which gradually disappears as the solution cools.

The temperature at which the color finally disappears from heated solutions of any of the colors given in the list depends on the rate of cooling. If cooled slowly the color will disappear at a much higher temperature than if cooled rapidly.

With the majority of the colors given in this list, a solution of sulphur dioxid in water will produce results similar to those obtained with the sodium bisulphite reagent. However, in working with a large number of coal-tar colors the latter reagent was found to give distinctive reactions in a larger number of cases than the sulphur dioxid solution. Preference was therefore given to the bisulphite solution as a reagent for general use.

THE DETERMINATION OF MOISTURE IN SIRUPS AND MOLASSES.

By W. D. HORNE.

The correct determination of water in sirups and molasses is very important both from the technical and the legal point of view—first, because this alone enables one to determine the true purity of the solution, and, secondly, because a sirup is defined by law as a solution containing not more than 25 per cent of water.

Various methods have been proposed for determining the water, but difficulties have been met with in all of them. Undiluted sirups dry slowly and frequently incompletely, necessitating prolonged heating or high temperatures, both of which tend to decompose organic matter. To dilute and mix with sand,

pumice, etc., solves this difficulty only partly. Vacuum drying is not always convenient. It necessitates elaborate apparatus and is often rather slow. The importance of finding a simple, quick, and accurate method for making this determination is apparent, and a very satisfactory procedure, gradually evolved by the writer in the course of many years of experimenting, is offered for the consideration of the association. The method reads as follows:

One gram of molasses is weighed into a flat-bottomed dish 3 inches wide and about 0.5 inch deep and containing a glass rod. About 0.8 cc of water is well mixed with the molasses and then about 30 grams of dry quartz sand, previously extracted with hydrochloric acid, is exactly weighed and added to the diluted molasses. The dish is placed on an open boiling water bath and stirred carefully and frequently during half an hour. This causes the free evaporation of about nine-tenths of the water at a moderate temperature. The dish is next placed in a water-jacketed air bath, where it is heated at the temperature of boiling water for two hours. After cooling it is weighed and reheated for one-hour intervals until the weight is constant.

This very easy method, requiring only the simplest devices and manipulations, gives, in from two to four hours, constant results that agree closely with determinations effected in partial vacuo during longer heating periods and under the influence of a fine stream of air dried by sulphuric acid. The method has been employed on refinery molasses for several years with great satisfaction.

In this work it is found that the degree Brix of the undiluted molasses plus the moisture determined as above amounts to 103.3 with great regularity, despite rather wide variations in the percentage of ash in the sirups.

Mr. Tolman submitted by title a report entitled "A study of the changes taking place in whisky stored in wood," by C. A. Crampton and himself. This report covers an investigation of eight years' duration conducted in the laboratory of the Bureau of Internal Revenue and is published in full in the Journal of the American Chemical Society for January, 1908, page 98. The conclusions reached are as follows:

(1) There are important relationships among the acids, esters, color, and solids in a properly aged whisky, which will differentiate it from artificial mixtures and from young spirit.

(2) All the constituents are undergoing changes as the aging process proceeds, and it is evident that the matured whisky is the result of these combined changes.

(3) The amount of higher alcohols increases in the matured whisky only in proportion to the concentration.

(4) Acids and esters reach an equilibrium, which is maintained after about three or four years.

(5) The characteristic aroma of American whisky is derived almost entirely from the charred package in which it is aged.

(6) The rye whiskies show a higher content of solids, acids, esters, etc., than do the Bourbon whiskies, but this is explained by the fact that heated warehouses are almost universally used for the maturing of rye whiskies, and unheated warehouses for the maturing of Bourbon whiskies.

(7) The improvement in flavor of whiskies stored in charred packages after the fourth year is due largely to concentration.

(8) The oily appearance of a matured whisky is due to material extracted from the charred package, as this appearance is almost lacking in whiskies aged in uncharred wood.

(9) The "body" of a whisky, so called, is due largely to the solids extracted from the wood.

DETECTION OF THICKENERS IN ICE CREAM.

By G. E. PATRICK.

The thickeners commonly used for ice cream to-day are gelatin, certain vegetable gums or jellies, and various forms of starch. Of the true vegetable substances gum tragacanth is most used, and it is believed that at least one other substance of this class is employed, but it was impossible to tell whether it was agar or some less common member of the group.

A test for the detection of such thickeners has been formulated which it is believed will prove useful if the conditions are closely observed, the main features of the method offering little that is new from a chemical point of view. Picric acid is used for precipitating gelatin, and alcohol for the gums. Preparatory to the test the liquid is clarified by coagulating the proteids with acid and heat and filtering. The details of the procedure, elaborated with the help of H. S. Bailey and B. McClelland, of the Dairy Laboratory, are as follows:

To 50 cc of ice cream add 25 cc of water, boil for half a minute to dissolve any thickener that may be present, add 2 cc of a 10 per cent solution of acetic acid, heat again just to boiling, add two or three heaping teaspoonfuls of kieselguhr, shake well, and filter immediately through a plaited filter. To 3 cc of the clear filtrate add 12 cc of 95 per cent alcohol and mix; this precipitates the milk proteids not removed in the clarification, together with the gums, and some of the gelatin, if much be present; add 3 cc of acidified alcohol prepared by mixing 95 cc of 95 per cent alcohol and 5 cc of concentrated hydrochloric acid; this dissolves the milk proteids completely. If the liquid is clear after this treatment, no gums or vegetable jellies are present. But, on the other hand, turbidity or a precipitate does not necessarily indicate the presence of a thickener; for this may be caused by the large amount of gelatin present or by eggs (more than three or four per gallon), or because the ice cream has become sour. Fortunately the precipitate due to gelatin or to the substance derived from eggs can be readily dissolved by a moderate dilution of the alcohol with water, for instance, by 3 cc of water in the test just described. This amount of water does not sensibly dissolve any precipitate due to vegetable gums or jellies, but entirely dissolves that due to gelatin and eggs. If gum tragacanth is present, the precipitate will be cohesive and stringy, or will become so upon shaking; while if the undissolved matter is due to other vegetable thickeners (possibly agar), it is finely flocculent and devoid of cohesive property.

If the ice cream is sour, there is sometimes a precipitate of another character, which must be derived chiefly, if not entirely, from the cane sugar present, since it appears very faintly, if at all, in tests upon unsweetened milks that have been allowed to sour. But this substance does not always develop in sour ice creams or in sweetened milks that have become sour; its appearance seems to depend upon some special property or condition of the milk, probably upon the presence of certain kinds of bacteria. Its nature is now being investigated, and Mr. C. A. Browne suggests that it may prove to be dextran. Whatever it may be, the precipitate which it yields with alcohol is not sensibly dissolved either by the acidified alcohol or by the water added to dissolve the gelatin and egg substance; therefore it must remain mixed with the vegetable gums and jellies. It does not resemble gum tragacanth, as it is not stringy, but does closely resemble the other vegetable thickener. It is therefore a serious obstacle in the test. Fortunately, however, its formation appears to be prevented by formaldehyde (experiments on this point are still in progress), and it is believed that if fresh ice cream is treated with a liberal dose of formalin there will be no trouble from this annoying substance even if the test is carried out several weeks later.

The test for the detection of gelatin was made on a small portion of diluted cream after boiling but before acidifying to precipitate proteids. The provi-

sional method of the association, originally published by Stokes (Analyst, 1837, p. 320), was used, which consists in clarifying the milk or cream with dilute mercuric nitrate solution and precipitating the gelatin in the filtrate with picric acid. It is in general a satisfactory test. But the interesting fact was observed by Mr. Bailey that in testing ice cream which had been sour for a week or more, or very sour milk, whether previously sugared or not, picric acid produces a yellow precipitate easily mistaken for that produced with gelatin. No way of distinguishing between the two has been found, but, as would be expected, formaldehyde prevents the formation of this "pseudo gelatin" and therefore samples thoroughly preserved with formalin present no difficulty from this source.

Starch, often used in a mixture with gum tragacanth, and sometimes alone, was detected in the usual way with iodine, using a portion of the boiled diluted sample.

REPORT ON THE DETERMINATION OF WATER IN FOODS.

By F. C. WEBER, *Associate Referee*.

The referee regrets that a full and definite report on this subject can not be given this year owing to lack of cooperation and of sufficient time to devote to the work. Circular letters were sent to 15 chemists asking for their collaboration; only 5 replied, stating that they could not take up the work this year.

The results obtained this year showed that drying in a vacuum of 0 to 5 mm of mercury over sulphuric acid compared favorably with drying in an oven with partial vacuum at 100° C. The results, however, in the vacuum oven were obtained in ten hours, while nine days were required to get the same results in the vacuum desiccator. The addition of phosphoric anhydride as an auxiliary drying agent in the vacuum-desiccator method did not appear to shorten the time of drying.

From the somewhat limited amount of work which has been done so far, the referee does not feel justified in condemning the method, though it is doubtful whether it could, even when modified, come into general use. The importance of further study on this subject is recognized, and it is therefore respectfully recommended that the work be continued next year.

THE CARBON DIOXID VALUE OF PURE COMPRESSED YEAST AND COMPRESSED YEAST AND STARCH COMPOUNDS.

By T. J. BRYAN.

The examination of numerous samples of compressed yeast in this laboratory showed that the majority of them contained added starch, usually either potato or corn starch.

The question arose, What is the value of the pure compressed yeast to the consumer as compared to the article mixed with starch? Some preliminary experiments were made, using samples of pure compressed yeast and yeast containing corn starch, such as were found on the market. In these preliminary tests the different yeasts were allowed to act upon a 10 per cent sugar solution and the volume of gas generated was measured. The results showed that the pure yeast had a greater carbon dioxide value than yeast mixed with starch. Owing to the size of the apparatus necessary for collecting and measuring the volume of the gas produced, it was thought better, in subsequent experiments, to determine the amount of carbon dioxide gas produced by the loss in weight. Furthermore, as the different yeasts tested were made by different manufacturers from different cultures, it was decided that the results secured were of little value in deciding the effect of the presence of starch in compressed yeast, and that samples of yeast from the same culture must be

secured, a portion of which should be kept in the pure state, and other portions mixed with potato and corn starch.

Seventy pounds of this pure yeast were mixed with 10 pounds of corn starch and 16 pounds of water (it was found necessary to add the water to make the mixing uniform and in order to produce a cake). To another portion of 70 pounds of the yeast were added 10 pounds of potato starch and 19 pounds of water. To the remaining portion of pure yeast no starch was added in the mixing. These three samples were then pressed separately and cut into 1-pound cakes which were each dipped in water before being wrapped, as is the custom of the manufacturer. These three samples were then allowed to act upon 100 cc of a 10 per cent sugar solution to which 75 cc of water had been added in an Erlenmeyer flask of about 190 cc capacity. This flask was provided with a calcium-chlorid tube adjusted by means of a cork in order to prevent loss of weight by evaporation. Two grams of yeast were used in each test and duplicates were run. The flasks were put in a box, the opening of which was covered with a towel and placed against the chimney where a temperature varying between 20° and 30° C. was maintained. During the last three days the box was replaced by a section of a bookcase and the temperature maintained ran a little higher than on the preceding days, varying between 25° and 30° C. Owing to the varying temperatures on different days only the results for the same periods on the same day are comparable. The flasks were weighed when they had been filled with the reagents and once every hour after that for ten hours, and again at the end of each twenty-four hours. Acknowledgments are due to Messrs. Nehls and Gardner and Mrs. M. B. Shulda for assistance in making the tests.

Table I shows the average loss in weight for the periods specified. Table II shows the average per cent of loss in weight (or the percentage of the weight of carbon dioxid gas produced for the specified periods), considering that the average weight of carbon dioxid produced by the pure yeast samples for each period is 100 per cent. It will be noted that after two hours the average loss in weight of the flasks containing pure yeast is, with a single exception, greater at every weighing.

The average percentages of the yield of carbon dioxid gas for twenty-four hours and for six hours, as given in Table II, show conclusively that the effect of starch upon yeast is to reduce the carbon dioxid value and that this percentage reduction is greater than the percentage of starch used in preparing the samples.

It has been claimed by some manufacturers that it is necessary to use starch in compressed yeast in order to preserve the same. The data in Table II show that on the fourteenth day the value of the pure yeast is greater than on the first day as compared with both of the starch yeast mixtures. The differences, however, are not sufficient, in the writer's opinion to justify the statement that the yeasts containing starch had deteriorated, though the data point in that direction. As fourteen days is a longer time than compressed yeast is kept before being put on the market and used the contention that starch is necessary to preserve yeast is seen to be absolutely false. Nineteen days after the yeast was prepared all of the samples were perfectly sweet.

It was thought desirable to test the action of these different yeasts in the making of bread. The bread was made with 650 grams of flour, 500 grams of water, and 10 grams of yeast or yeast starch mixture. On the second day pure yeast yielded bread having a volume of 2,225 cc; corn-starch yeast mixture, bread 2,100 cc in volume. On the fifth day pure yeast yielded bread of a volume of 2,000 cc; corn-starch yeast yielded bread 1,860 cc in volume. On the twelfth day pure yeast yielded bread 2,150 cc; corn-starch yeast mixture, bread 2,015 cc. On the thirteenth day pure yeast yielded bread 3,000 cc in volume and corn-starch yeast mixture, bread of 2,575 cc. On the fourteenth day the

bread from the pure yeast had a volume of 2,820 cc, while the corn-starch yeast mixture bread had a volume of 2,650 cc.

As in the action of yeast on sugar solutions, the actual baking tests show that the carbon dioxide value is always greater in the case of pure yeast. By comparison it will be found, however, that the average percentage difference in the size of the loaves is less than the average difference in loss of weight of the sugar solutions for the same periods. This is due to the fact that in the case of the bread it is only the final volumes that are compared, whereas to make the results comparable with those obtained by the action of yeast on sugar solutions it would be necessary to determine the difference in volume (for 2 grams of yeast) between the freshly mixed bread and the final volume of the loaf.

There is much work to be done before the action of starch in yeast will be known in all its details. The writer feels, however, that the results here given are sufficient to justify the statement that starch in compressed yeast is an adulteration. The advantage to the manufacturer is easily seen when we consider that he sells starch which costs less than 3 cents per pound at the price of yeast which costs 15 cents per pound. A standard for compressed yeast that will exclude the use of added starch is therefore most desirable. Next to the quality of the flour the quality of the yeast is of prime importance to the 70,000,000 people of this country in the preparation of their bread.

TABLE I.—Average loss in weight of sugar solution containing pure yeast and yeast-starch mixtures, September 19 to October 2, 1907.

Number of hours.	1 day old.			2 days old.			5 days old.		
	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.
	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>
1	0.035	0.012	0.028	0.039	0.013	0.019			
2	.081	.045	.083	.071	.042	.055	0.027	0.034	0.033
3	.185	.127	.170	.193	.129	.150	.089	.082	.091
4	.325	.232	.268	.314	.216	.239	.300	.246	.239
5	.598	.413	.437	.524	.372	.360	.404	.318	.375
6	.770	.610	.631	.664	.495	.519	.587	.505	.572
7	.888	.719	.757	.849	.678	.687	.824	.665	.754
8	1.064	.874	.920	1.008	.819	.795	.916	.753	.873
9	1.222	1.022	1.075	1.136	.914	.903	1.072	.884	.995
10	1.329	1.115	1.170	1.206	1.003	1.011	1.181	.972	1.100
24	2.779	2.345	2.415	2.661	2.189	2.313	2.520	2.230	2.378
	6 days old.			7 days old.			8 days old.		
1	.047	.051	.031	.018	.023	.037	.051	.047	.044
3	.163	.148	.126	.158	.120	.146	.158	.130	.127
4	.373	.315	.280	.268	.200	.217	.325	.233	.252
5	.601	.517	.452	.533	.409	.461	.525	.410	.409
6	.742	.616	.613	.748	.570	.645	.695	.522	.542
7	.982	.817	.797	.930	.710	.789	.850	.662	.683
8	1.098	.915	.907	1.082	.829	.915	1.025	.798	.826
9	1.228	1.026	1.024	1.202	.937	1.027	1.129	.878	.916
10				1.326	1.037	1.123	1.266	.987	1.025
24	2.645	2.334	2.279	2.662	2.167	2.392	2.517	2.041	2.167
	9 days old.			13 days old.			14 days old.		
1	.034	.041	.029	.039	.039	.031	.022	.016	.019
2	.193	.174	.123	.199	.131	.106	.127	.086	.088
3	.408	.376	.332	.391	.240	.254	.279	.188	.197
4	.642	.558	.492	.631	.442	.458	.513	.375	.398
5	.813	.690	.631	.816	.593	.600	.721	.537	.564
6	.869	.796	.768	1.015	.750	.771	.885	.674	.707
7	1.119	.904	.912				1.093	.847	.883
8	1.323	1.055	1.043				1.218	.950	.988
9	1.469	1.170	1.164						
21				3.120	2.460	2.517			
24	2.970	2.522	2.392				3.134	2.546	2.655

TABLE II.—Carbon dioxid gas produced in sugar solutions containing yeast-starch mixtures as compared with those containing pure yeast, rated at 100.

Age of yeast.	For twenty-four hours.			For six hours.		
	Pure yeast.	Potato starch mixture.	Corn-starch mixture.	Pure yeast.	Potato starch mixture.	Corn-starch mixture.
<i>Days.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	100	84.4	86.9	100	79.2	81.9
2.....	100	82.3	86.9	100	74.5	78.1
3.....	100	88.5	94.4	100	86.0	97.4
4.....	100	88.2	86.2	100	83.0	82.6
5.....	100	81.4	89.9	100	76.2	86.2
6.....	100	81.1	86.1	100	75.1	77.9
7.....	100	84.9	80.5	100	91.6	88.4
8.....	100	78.8	80.6	100	73.9	76.0
13.....	100	81.2	84.7	100	76.2	79.9
14.....	100			100		
Average.....	100	83.4	86.2	100	79.5	83.1

Vice-President Snyder took the chair at this point and Mr. J. P. Street delivered the annual presidential address.

PRESIDENT'S ADDRESS.

By J. P. STREET.

Gentlemen of the Association: An unfortunate unwritten law, supported by the custom of many years, demands an address from your presiding officer. My 23 predecessors have covered the ground so well, and have appreciated the merits and the failings of this association so thoroughly, that there is but little left for me to do save to repeat their congratulations and emphasize their warnings.

That the original object of this association was to secure uniform and accurate methods for the analysis of fertilizers is known to you all. Likewise you are all familiar with our steady growth, until now we include within the province of our study and control practically all materials connected with agricultural industry. This widening of the association's activities has limited in some degree the interest of the members in its work as a whole. This is an inevitable result of our expansion, and, deplorable as it is from certain points of view, it could not be expected that either time, inclination, or immediate interest would lead all the members to take part in all the work. This is no reason, however, why every member should not take part in some of the cooperative work each year, and by his careful work help in the solution of the numerous problems that still confront the agricultural chemist. In the association's earlier days it was the custom for practically every institution represented in its membership to share in the cooperative work, and this was easily possible when fertilizers alone were the subject of study. It was also the custom then that this work should be performed by the experienced men of the staff, so that when the results were presented to the referee for comparison and study they would furnish a reasonable test of the method's accuracy, and not of the capability of the analysts. But in later years this condition to a great extent changed; the official samples all too often were used to check the younger assistants rather than the method itself. This resulted in certain cases in injustice to methods which proved later to be thoroughly dependable, and it would seem that the publication of certain of the comparative results must have had a harmful effect on our standing as an association. It is a matter of deep gratification,

therefore, to observe that as a result of recent agitation of this subject the pendulum is swinging back, and we find that a large proportion of our association work is again intrusted to those best qualified to handle it. While the privilege of assisting in our work should be denied to none of our members, still greater efficiency would be secured if even wider latitude were permitted the referees in the selection of their associates. These associates should be not only skilled manipulators, but should have given particular attention to the subject under investigation. While general cooperation, as I shall point out later, is most desirable, it would be far better for the skilled chemist to decline to take part in an investigation than to intrust the details to an inexperienced assistant. Surely there is no member of this association who is not expert in at least one branch of agricultural analysis and whose work would not be of great value if properly directed.

As a matter of interest, I have tabulated the work of the past ten years, to determine the amount of cooperation of the various State laboratories. In preparing this summary I have naturally left entirely out of consideration the Bureau of Chemistry, and it is only just that attention should be called to the valuable work done by the Bureau each year in the study of our methods. With five referees and eight associates included in its organization this year, besides doubtless many others assisting in the work of other referees, the interest of its controlling officers in this association is manifest, and we are under deep obligation to them for the liberal policy which they have fathered. When we consider the results of my tabulation, and learn what the attitude of the various State institutions has been toward this association, a different story is told. I find that two of the States took part in the work in each of the ten years, thirteen in from seven to nine years, fourteen in from four to six years, three in from one to three years, and four States gave no assistance whatever. Four States, three of them distinctively agricultural, showed not the slightest practical interest in the work of the association during a period of ten years. By further subdividing the time under consideration into two periods of five years each, it is seen that there has been an increased activity in all the groups of States during the past five years, with the exception of the Middle West, this activity being specially marked in the Central and Pacific States. The encouragement furnished by this second classification is somewhat diminished, however, by the fact that from 1901 to 1906, inclusive, six States cooperated but twice, thirteen but once, and nine not at all. Do not these facts, which can be easily verified by a study of our reports, show either that there is a lack of sympathy with the association and its work on the part of those in control of many of our experiment stations, or that there is a willingness to cast the burden of this work on others, and enjoy the fruits of their labors. Efficient and accurate methods of analysis are the groundwork of all agricultural research, and any station which fails to aid in their development and application shirks a manifest duty and plays the rôle of a parasite on this association.

There is another subject which I feel many of our members do not approach with a keen sense of the duty involved. In 1896, when acting as referee on nitrogen, I made a recommendation, which after much discussion by the committee was distorted to read as follows: *Resolved*, That it is the sense of this association that the forms of the nitrogen in commercial fertilizers should be reported, where it is possible to do so." The clause "where it is possible to do so" cast the resolution, as far as its effect was concerned, into

"A limbo large and broad, since called
The paradise of fools."

In 1902, as chairman of Committee A on Recommendations, I was able to rescue the matter in a measure from its undeserved oblivion by urging the association to take a somewhat more decided stand by the adoption of the following resolution: "That it be earnestly urged that, in reporting the analyses of complete fertilizers, the amounts of nitrogen existing in the form of nitrates, ammonia salts, and organic nitrogen be specifically stated." And there the matter rests to-day.

In spite of this specific recommendation from what should be considered the court of last resort in matters pertaining to agricultural chemistry, what has been the attitude of those having charge of the thirty-one different fertilizer controls? Five controls report the three forms of nitrogen, three report nitrogen soluble and insoluble in water, or total nitrogen and nitrates, and one reports total and available nitrogen; twenty-two report only total nitrogen. Fifteen of these delinquents report phosphoric acid in three forms, and four of them separate the potash, as from chlorid and sulphates. Is not this a glaring inconsistency? The form of the nitrogen, the most important and most expensive ingredient of the fertilizer, is absolutely ignored by 70 per cent of the inspection officials, while insoluble phosphoric acid is carefully determined by most of them by a method whose accuracy is universally discredited. Surely there is no excuse for this careless, I will not say ignorant, attitude. Excellent, exact, and rapid methods for the determination of nitrates and ammonia salts are at the disposal of every inspection official, and the resulting benefits would more than compensate for the slight additional time consumed.

The only excuse that I have ever heard offered for failure to make these determinations was lack of time to do more than the law required. I can not believe this an honest excuse. Is it not time that the fertilizer appropriation or the income from fertilizer fees should cease to be considered as a financial aid to carry on more interesting investigations in other lines of work? Is it not our official duty, not simply to make a perfunctory examination to satisfy the requirements of the law, but also to impart to the consumer all the information possible from our present methods of analysis as to the sources of the materials used in compounding the mixtures offered to him for purchase? And what has been the result of this indifference, this neglect of duty, this shirking of responsibility? The fertilizer manufacturer has watched us, gentlemen; he has learned the weak points in our armor, and he has always kept a decade in advance of us. The commercial instinct is not dormant in his heart. The high price of nitrogenous fertilizing materials has tempted him to use any and all materials, often inert and valueless, for he knew that with few exceptions the inspection officials would not differentiate these from higher grade materials, but would by their analysis classify them with the best and most available forms, and give them a correspondingly high commercial valuation. Is it any wonder, then, and are we entirely blameless, that the use of these inert materials is continually increasing, until now we are forced in defense of the interests confided to our care to attempt some laboratory method for distinguishing available and inert organic nitrogen? For several years the association has been studying methods with this end in view, but the progress has been slow and the cooperative work has been slight. It is a subject which merits the earnest thought and the patient study of every fertilizer control official, and without this concentrated effort the problem will long remain unsolved. Our obligation to the manufacturer who continues to use only high-grade materials in his fertilizers is as well defined as is our duty toward our constituents, the agricultural public, who

have a right to know the nature of the materials offered them as possessing high fertilizing value.

A review of the history of this association clearly indicates that it is a conservative organization. This conservatism has always been an element of strength, and the wisdom of our attitude toward the adoption of new methods can not be questioned. When we have been confronted by new agricultural problems, however, it must be confessed that in some cases at least our conservatism has degenerated into a sort of artful dodging. Two queries well illustrate my contention: How shall we measure the availability of basic slag? And is there such a thing as "available" potash as distinguished from water-soluble potash? Our attitude toward these problems is known by you all. Basic slag has been the shuttlecock of this association for a number of years, and, as far as any official pronouncement as to its status and methods to determine its worth is concerned, we are just about where we started. The importance of this material as a phosphatic fertilizer will increase with the years, and the time is not far distant when we shall be obliged to decide the question by some other method than skillful side stepping. The question of "available" potash is being forced on our attention by the manufacturers, who make the twofold claim that our present methods do not credit them with all the water-soluble potash they supply in their mixtures and do not recognize the agricultural value of certain forms of potash which are insoluble in water. The work of our referee on potash for the past two years seems to support the contention that not all the water-soluble potash added appears in the analysis when made by the official methods. It is true that we may have serious doubts as to the wisdom of adopting any modification of the method which permits the use of a solvent, which might bring inert potash into solution, and I believe the association made no mistake in rejecting the recommendation of the referee for the adoption of this modification. Nevertheless the manufacturer, who surely is entitled to justice and protection quite as much as the consumer, makes a claim which our own work seems to justify, and the obligation rests upon us to seek some modification of our methods which will remove from them even the slightest stigma of injustice. And the same holds true with the question of "available potash." We are not in a position to affirm or deny the manufacturer's claim that the potash which he adds in an organic or mineral form, and which is not soluble in water, possesses an agricultural value approximately the same as the water-soluble form. The claim, however, demands our consideration and is worthy of our careful study.

We must not deceive ourselves into the comforting belief that the labors of this association are at an end. In spite of the remarkable progress made by our very efficient referees in the last few years in perfecting our methods for food analysis, every food chemist realizes the gaps which still exist in these methods and the new problems that are constantly being presented. The analysis of drugs is almost an unexplored field. Your attention has just been called to some of the unsolved problems in fertilizer analysis. A brief glance at the subject of cattle foods will show that here is another unexhausted territory. It is hardly necessary to call attention to the fact that no one of the ordinary determinations in cattle foods, with the possible exception of ash, is scientifically accurate. These inaccuracies are, however, in themselves of minor importance from a feeding standpoint, except that the algebraic sum of all the errors involved, by our present method of calculation by difference, falls upon the "nitrogen-free extract." Not only is the aggregate percentage conceded to "nitrogen-free extract" usually inexact, but it is manifestly unscientific to group together under one heading such dissimilar substances as

sugars, starch, gums, pectin bodies, pentosans, lignin, and the various modifications of the hemicelluloses and oxycelluloses, especially when they constitute such a large part of the feed under examination, varying from 26 per cent in cotton-seed meal to 80 per cent in rice. The meaninglessness of this term has been recognized by all, and in recent years much excellent, and in some cases conclusive, work has been done toward the separation and determination of many of these carbohydrate bodies. Recent work of my own in a study of the carbohydrates of dried beet pulp has convinced me that this matter is important not only from an analytical point of view, but also in that its solution would shed much light on many of the problems of animal feeding. The sugars and starches are by common consent rated as the most assimilable forms of the carbohydrates, and yet feeding experiments have shown that beet pulp, which contains these forms in minimum quantities, was more efficacious than hominy meal, a typical starchy feed. Does not this surprising result indicate either that our methods of feed analysis are misleading, or that certain of the carbohydrates generally classed as inferior for feeding purposes are, in certain feeds at least, of superior quality and of high digestibility?

By subjecting beet pulp successively to a series of solvents, as originally suggested by Dragendorff, and further developed by Parsons, Frear, Browne, Sherman, and others, and analyzing the various residues and solutions, I secured some very interesting and suggestive results. Large amounts of araban, galactan, lignin acids, and lignin were found, with smaller quantities of pectin, parapectin, and reducing and invert sugars; while nearly one-quarter, or 16 per cent, of the carbohydrates existed as unidentified hemicelluloses, which were dissolved by dilute sulphuric acid and were present in an incompletely hydrolysed state. It was shown that the galactan was present in three forms; one fraction was soluble in 80 per cent alcohol, one was soluble in cold water, and the balance was hydrolysed by dilute sulphuric acid. The pentosan arabans in this case were even more complex, and varied greatly in their resistance to solvents. Varying portions were soluble in cold water, in dilute acid, in warm water, in diastase solution, and in dilute alkali, and still another portion was removed by chlorination. The chlorination process gave a percentage of cellulose 6.5 per cent lower than the official method, the residue being entirely free of pentosans and proteids, but still containing one-fourth of the original ash.

Time does not permit me to go into further details as to the results of this investigation. My experience with this material, however, leads me to the conclusion that for a rational basis of scientific feeding we must not be content with the determination of the total of any ingredient, but must differentiate its various forms as indicated by their behavior with a series of solvents. The fact that we have in beet pulp apparently at least six different modifications of araban and three of galactan would encourage the assumption that a determination of total araban and total galactan in a feed conveys as little useful information as one of total phosphoric acid in a fertilizer.

On reading the constitution of this association I note that its objects are two-fold, not only to secure uniform and accurate methods of analysis, as already mentioned, but also "to afford opportunity for the discussion of matters of interest to agricultural chemists." It would seem that this second and very important object of the association had been to a great extent neglected. We have increased the scope of the association without a corresponding increase in the length of our meeting, with the result that practically all of our time has been occupied in the presentation of reports, too often presented in an undigested form, which because the necessary data are not before us, are unin-

telligible to all except the few who have taken part in the work on the particular subject in question. Discussion of the reports has been reduced to a minimum; in fact, it has been discouraged. As a result, our meetings have lost some of their interest, and the benefits resulting from them have somewhat diminished. No member of this association, I am confident, will deny that one of the greatest advantages derived from attendance at these meetings is the opportunity for the exchange of ideas, whereby we learn the experience of others in the lines of work that have presented difficulties to us ourselves. This information we have been forced to obtain outside the formal meetings of the association, and the benefits accruing therefrom have therefore been limited to those whose intimate acquaintance has permitted questions a stranger would not feel at liberty to ask. It would seem that these benefits derived from experience should be open to all our members, and that the proper place for their discussion is the floor of this convention.

It is impossible for us all to take an equal interest in all the ramified work of this association, and a general meeting for papers and discussion might not meet with unqualified success. I would urge, therefore, that one day be devoted entirely to the reading of papers and their discussion, and that for this purpose the convention be divided into three sections covering the same subjects as our present committees on recommendations. All papers to be read in these sectional meetings should be referred to the proper committee with full power to reject the same, or assign places to them in the programme, which should be sent to all members prior to the convention. I believe in this way, gentlemen, that the interest in our meetings would be greatly intensified, and an immediate as well as lasting benefit conferred upon our members.

The constantly changing personnel of this association is an element of weakness and gives to it a character of impermanency. This fact has been particularly impressed upon me during the past two years in my work with the committee on revision of methods. We were confronted by a great mass of methods couched in as many forms of English as there had been referees, with varying nomenclature and with no fixed or systematic method of treatment. Under the conditions attending the origin of our methods no other result could have been expected. Your committee has attempted, and I trust with a considerable degree of success, to bring order out of this chaos and has rewritten the methods with the idea of clearness and uniformity of statement constantly in mind. And yet all this labor will go for naught and our methods in a few years will return to their previous state of indefiniteness unless our present system is corrected.

The one permanent office in this association has been that of secretary. There seems to be an unwritten law that during his natural life Doctor Wiley shall be our secretary, his occupancy of that office being conditioned only by his good behavior. The benefits accruing to the association by this, its one permanent feature, are undoubted, and any effort to insure further permanency in other departments of the association would seem worthy of encouragement. In order, therefore, to launch the association upon a settled policy in the matter of methods the primal reason for our existence as an association I strongly recommend the appointment of a permanent committee on methods, consisting of nine members, who shall hold office until disqualified by the provisions of the constitution; this committee to consist of three subcommittees corresponding to our present committees A, B, and C. This committee shall receive all reports of referees at least three weeks before the annual meeting of the association, should edit them for presentation to the convention, and after the adoption of new methods or modifications of the old ones, should incorporate

them in our official methods in harmony with our newly established system of treatment. In this way the policy of the association as regards methods would continue unaltered year after year, and the committees would not labor under the disadvantage, so often evidenced in the past, of unfamiliarity with the work of their predecessors.

One more presidential suggestion, and I am through. Our constitution provides in definite terms who are eligible to membership in this association. Section 2 reads as follows: "Analytical chemists connected with the United States Department of Agriculture, or with any State or National agricultural experiment station or agricultural college, or with any State or National institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership." And later in the same section we read: "All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote." It is not entirely clear in my mind just what the words "any State or National" mean. I had supposed that our membership was limited to agricultural chemists in this country, but the above wording, according to my interpretation, might include those connected with agricultural institutions in any State or country. It hardly seems possible that this could have been the meaning of the framers of our constitution, and but for the fact that our Canadian confrères have always enjoyed all the privileges of the association, I would not raise the point at this time. We all appreciate the valuable contributions to our methods from our Canadian friends, and I think no one would deny to them the privileges of membership, and yet I believe we should maintain this as a North American association. I would suggest, therefore, that this ambiguity in the constitution be considered at this meeting by a special committee, and would recommend that the constitution be so amended as to include Canada and Mexico as the only foreign countries entitled to representation.

The constitution provides that unofficial analytical chemists are not "entitled to enter motions or vote." Surely the more important duty of acting as referee should also be denied them, and yet they are not specifically barred from this privilege by the constitution. Unofficial chemists have frequently acted as our referees in the past, and in many cases the association has benefited by their labors; and yet, with all respect to these gentlemen, the precedent established is a dangerous one and can not be too speedily corrected. We should heed Virgil's warning: "Timeo Danaos et dona ferentes."

I would not have the members of the association assume from the somewhat critical tone of my remarks that I am unmindful of the excellent work of the past, of the authoritative position the association now occupies in scientific and legal circles, and of the patient, self-denying, self-effacing work of many of our members in advancing agricultural analysis to its present high plane. But self-complacency and self-congratulation are not incentives to determined, progressive action. Much of our work in the past has had to do with the comparatively simple problems of agricultural analysis; the work of the future will demand effort of an even higher grade, based on careful, painstaking, intelligently directed research. Every station owes it to the public to do some work of original investigation. The analytical methods employed are in a sense the tools by which any agricultural chemical investigation is made possible. The perfection of these tools alone makes possible successful investigation with them. It is certainly, then, no unworthy or unimportant work for a station to spend time in the careful study and perfection of methods.

APPOINTMENT OF COMMITTEES.

At the close of the president's address, which was received with marked approval by the association, the following committees were announced:

Committee on resolutions: Messrs. Van Slyke, Hopkins, and Withers.

Committee on nominations: Messrs. Hartwell, Bartlett, and Cathcart.

Committee on amendments to the constitution: Messrs. Frear, Kilgore, and Lipman.

On motion by Doctor Wiley the recommendations offered in the president's address were referred to the committee on resolutions.

The report of the referee on nitrogen was then presented by the secretary as follows:

REPORT ON THE DETERMINATION OF NITROGEN.

By CHARLES L. PENNY, *Referee*.

The following circular letter, together with samples of corn meal and cotton-seed meal, was sent to members of the association:

The referee and associate referee on nitrogen request you to determine the nitrogen and moisture in a sample of corn meal and one of cotton-seed meal sent to you by mail.

It is requested that nitrogen be determined by the Kjeldahl method and also, if convenient, by the soda-lime method or the absolute method. As a comparison of analytical figures is contemplated, with a view to repetition in cases of wide variation from the average, it is hoped that the work may be done promptly.

Please state in reference to the moisture and the Kjeldahl method—

- (1) Temperature at which moisture is determined, and also whether in air or hydrogen.
- (2) Duplicates of nitrogen separately, not merely the average.
- (3) Whether at end of digestion the hot sulphuric acid was quite colorless.
- (4) Whether permanganate of potash was used or not; and if used, whether to the point of permanent coloration.
- (5) Any other reagents used in the digestion.
- (6) The quantity of substance taken for a determination, the amount of sulphuric acid used, and the size of digestion flasks.
- (7) The approximate time of digestion and of distillation.
- (8) Whether blanks were digested and distilled; and if so, the amount of correction; also whether the distillation was proved to be complete by the collection of a second portion of distillate.
- (9) Method of standardizing acid and the indicator used.

It is hoped that all chemists will cooperate in this work, as it is believed that no single determination interests so many as does that of nitrogen, that few are so important, and that unfortunately variations in analytical results have been unreasonably wide.

Address replies to the referee—

C. L. PENNY,
Agricultural Experiment Station, Newark, Del.

FEBRUARY 16, 1907.

Fifty-three analysts took part in the work, contributing a valuable collection of opinions and experiments. The generous portion of time and labor given by them shows a deep interest in every attempt at the improvement of our analytical methods. Over 420 separate determinations of nitrogen and about 120 of moisture were made, some according to strictly prescribed methods and others according to methods deviating in several respects from these. The list of analysts participating in this work may be found in the table of moisture

determinations. The samples were taken from a quantity of material thoroughly mixed, bottled, and sealed; it is believed that they were as nearly uniform as they could be made, and fully secured from change in moisture content or other change. The following table shows the percentage of moisture found by the several analysts and the methods pursued by each:

TABLE I.—*Results of cooperative work on the determination of moisture in corn meal and cotton-seed meal.*

Analyst.	Corn meal.		Cotton-seed meal.		Method.
	Results.	Average.	Results.	Average.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
Asbury, S. E.....					
Atkinson, F. C.....	{ 13.45 13.45 }	13.45	{ 7.75 7.60 7.75 }	7.70	Air; 99°-100° C.
Bailey, E. Monroe					
Barber, Kate E.....					
Blair, A. W.....	{ 13.42 13.35 13.55 }	13.39	{ 7.70 7.85 7.77 }	7.78	{ Vacuum; three and one-half hours; 70°-75° C., 2 grams of substance.
Bradley, C. E.....	{ 13.52 14.41 14.43 }	13.54	{ 7.77 8.69 8.95 }	7.77	Thirty-six hours; 96° C. (water).
Do.....		14.42		8.82	Twelve hours; 100° C. (air).
Carlyle, E. C.....					
Crocker, C. S.....	{ 14.15 13.87 }	14.15	{ 8.00 7.67 }	8.00	Air bath; 212° F.; five hours.
Cruse, J. C.....	{ 13.62 15.19 15.15 }	13.75	{ 7.91 8.98 9.04 }	7.79	
Edmond, H. D.....		15.17		9.01	{ Hydrogen; temperature boiling water; Corn meal—2.981 grams, 2.847 grams; cotton-seed meal—3.071 grams, 2.436 grams.
Fetzer, L. W.....	{ 13.34 13.42 12.14 }	13.38	{ 8.39 8.41 6.71 }	8.40	
Ford, A. G.....	{ 12.17 13.15 13.10 }	12.16	{ 6.60 7.48 7.47 }	6.66	Air; boiling water oven.
Fuller, F. D.....	{ 13.94 13.97 13.93 }	13.13	{ 8.05 8.04 8.18 }	7.47	{ Air; 98° C.; five hours; 2 grams substance.
Fulmer, H. L.....		13.95		8.09	{ Hydrogen; 210° F. (98.8° C.); 3 grams substance.
Gascoyne, W. J.....					
Grainger, W. E.....	{ 13.18 12.03 12.21 12.16 }		{ 7.64 7.63 7.63 7.63 }	7.64	Air; water oven; 98° C.; five hours.
Do.....		12.65			
Greaves, J. E.....	{ 12.67 12.45 }	12.56	{ 6.13 6.15 }	6.14	Air; water oven; to constant weight.
Green, H. L.....					
Hall, J. A., jr.....	{ 12.45 12.50 }	12.48	{ 6.80 6.90 }	6.85	{ Air; temperature boiling water; ten hours; 2 grams substance.
Halligan, J. E.....					
Hammond, H. S.....					
Hare, R. F.....	{ 13.13 13.53 14.88 15.10 }	13.33	{ 7.38 7.00 9.12 9.08 }	7.19	Hydrogen; 96° C.
Jones, J. Shirley.....	{ 13.03 13.22 13.12 }	14.99	{ 7.24 7.46 7.49 }	9.10	Air; 100°-101° C.
Kerr, Robert H.....	{ 13.69 13.69 13.96 13.96 }	13.03	{ 7.24 7.46 7.91 7.92 }	7.24	Air; water oven; five hours.
Knisely, A. L.....		13.17		7.48	Air; oven; eighteen hours; 94°-96° C.
Do.....		13.69		7.92	Eighteen hours longer, 98° C.
Kunst, F. B.....	{ 13.96 13.96 13.97 14.25 14.19 14.23 }	13.96	{ 7.36 7.34 8.40 8.43 8.45 8.45 }	7.35	{ Vacuo-steam bath; temperature of boiling water; five hours; 2 grams.
La Shell, L. L.....	{ 13.70 12.87 13.70 13.70 }	14.22	{ 7.74 7.23 7.54 7.55 }	8.43	Air; 100° C.; three hours.
Macy, E. J.....	{ 13.38 13.70 12.08 12.49 }	13.70	{ 7.62 7.75 7.07 6.72 }	7.05	{ Vacuum; three and one-half hours; 2 grams; 70°-75° C.
Do.....		12.87		7.23	Water oven; ten hours.
McDonnell, C. C.....	{ 13.70 13.70 13.38 13.70 }	13.70	{ 7.54 7.55 7.62 7.75 }	7.55	{ Air; water oven; temperature of boiling water; five hours.
Mitchell, S. R.....	{ 12.08 12.49 13.65 13.71 }	13.54	{ 7.07 6.72 7.85 7.83 }	7.69	{ Air; 96° C.
Morgan, J. F.....	{ 14.30 14.35 }	12.29	{ 6.90 6.72 7.85 7.83 }	6.90	{ Hydrogen; 96° C.
Do.....		13.68		7.84	{ Hydrogen; five hours; 2 grams; steam.
Do.....		14.33		8.36	Hydrogen; ten hours; 2 grams.
Do.....					Hydrogen; thirteen hours; 2 grams.

TABLE I.—*Results of cooperative work, etc.*—Continued.

Analyst.	Corn meal.		Cotton-seed meal.		Method.
	Results.	Average.	Results.	Average.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
Morgan, J. F.....	{ 14.31 14.39 }	14.35	{ 8.41 8.39 }	8.40	Hydrogen; sixteen hours; 2 grams.
Do.....	14.36	14.36	{ 8.81 8.70 }	8.76	{ Paraffin oven; twenty hours; 2 grams; 95° C.
Do.....	14.36	14.36	{ 8.86 8.71 }	8.79	{ Paraffin oven; twenty-three hours; 2 grams; 95° C.
Norton, J. H.....	{ 13.03 12.96 }	13.00	{ 7.64 7.30 }	7.47	Air; 110° C.; two hours.
Do.....			{ 7.36 7.70 }	7.53	{ Air; 110° C.; three and one-half hours.
Ogden, A. W.....	13.07	13.07	7.42	7.42	Air; 100° C.; three hours.
Do.....	13.07	13.07	7.47	7.47	Air; 100° C.; five hours.
Do.....	13.13	13.13	7.43	7.43	Air; 100° C.; two hours.
Do.....	13.15	13.15	7.27	7.27	Air; 100° C.; two and one-half hours.
Do.....	15.16	15.16	8.75	8.75	Hydrogen; 100° C.; two hours.
Do.....	15.30	15.30	9.04	9.04	Hydrogen; 100° C.; four hours.
Do.....	15.06	15.06	8.63	8.63	Hydrogen; 100° C.; three hours.
Pappe, T. F.....	{ 11.88 11.79 }	11.84	{ 6.63 6.63 }	6.63	{ Air; water oven; temperature of boil- ing water.
Parkens, J. H.....					
Patten, A. J.....	{ 13.45 13.43 }	13.44	{ 7.81 7.67 }	7.74	
Rather, J. B.....					
Robb, J. B.....	{ 11.96 11.94 }	11.95	{ 6.80 6.92 }	6.86	Air; five hours.
Do.....	{ 12.53 12.44 }	12.49	{ 7.17 7.08 }	7.13	Hydrogen; five hours.
Do.....	{ 13.52 13.43 }	13.48	{ 7.75 7.70 }	7.73	Air; twenty-one hours.
Roberts, George.....					
Shanley, E. J.....	{ 14.99 15.04 }	15.02	{ 9.41 9.51 }	9.46	{ Hydrogen; temperature of boiling water.
Shedd, O. M.....	{ 12.79 12.61 }	12.70	{ 7.59 7.44 }	7.52	Air; 96½° C.; five hours.
Do.....	{ 12.75 12.75 }	12.77	{ 7.59 7.48 }	7.54	Air; 96½° C.; to constant weight.
Do.....	{ 14.41 14.44 }	14.43	{ 8.65 8.76 }	8.71	{ Natural gas; water oven 96½° C.; five hours.
Do.....	{ 14.68 14.71 }	14.70	{ 8.93 8.92 }	8.93	{ Natural gas; water oven; 96½° C. to constant weight.
Sheib, S. H.....	13.32	13.32	7.55	7.55	Air; water bath; five hours.
Smith, P. H.....	{ 14.02 13.95 }	13.99	{ 8.30 8.19 }	8.25	Air; 100° C.; nineteen hours.
Spears, H. D.....					
Summers, J. C.....	{ 12.65 12.68 }	12.67	{ 6.94 7.06 }	7.00	Air; water oven; 98° C.; eight hours.
Taggart, W. G.....					
Thatcher, R. W.....	{ 14.66 14.68 }	14.67	{ 9.13 8.92 }	9.03	{ Hydrogen; Caldwell tubes; eight hours.
Trescot, T. C.....	14.93	14.93	8.78	8.78	Vacuum oven; 96° C.; nine hours.
Watkins, H. R.....	{ 14.04 13.97 }	14.01	{ 8.18 8.17 }	8.18	Water oven; twelve hours.
Whittier, A. C.....			{ 8.54 8.43 }	8.43	Air; 97° C.
Wiley, R. C.....	{ 11.77 11.70 }	11.74	{ 6.72 6.73 }	6.73	Air; water oven; 100° C.
Williamson, C. S.....	12.80	12.80	7.38	7.38	Air; 100° C.; four hours.
Wilson, R. N.....	{ 13.23 13.22 }	13.23	{ 7.10 7.16 }	7.13	{ Vacuum; 70°-75° C.; three and one- hours; 2 grams.
Mean of all different determinations.		13.54		7.82	
Maximum.....		15.30		9.46	
Minimum.....		11.74		6.14	

The moisture was determined in a variety of ways, and there is considerable divergence in the results, partly due to difference in the method and partly to the analysts, but, it is believed, due only slightly to actual differences among the samples. The fluctuations in the moisture of the corn meal, 3.56 per cent, are nearly one-fourth and in that of the cotton-seed meal, 3.332 per cent, over one-third of the actual moisture in the respective substances.

Two well-known and important facts appear from the comparative work of some of the analysts, namely, the oxidizing action of the air, which sometimes accounts for a loss of over 2 per cent of moisture as compared with a determination in hydrogen and the great influence exerted by the length of time allowed for drying. While it may not, perhaps, be within the scope of a report on determining nitrogen, I would emphasize the advantage of returning to a four or five hour time limit for moisture determinations in feeds and similar products. The figures of Mr. Morgan and Mr. Knisely show that even ten or eighteen hours of heating do not give constant weight. Probably every feed control laboratory establishes its own arbitrary time limit, but it would be better if a fixed temperature and a time limit were prescribed to provide against variations of the boiling point at different altitudes.

The following table shows the percentages of nitrogen as determined by 52 analysts, together with the various methods pursued. The numbers assigned are arbitrary and do not refer to the preceding table:

TABLE II.—*Percentages of nitrogen found in corn meal and cotton-seed meal, based on the air-dry, not the absolutely dry, state.*

(a) SULPHURIC ACID METHODS.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
1.....	{ 1.475 1.465 }	1.47	{ 6.797 6.800 }	6.799	{ Digested two and one-half hours; colorless; green with KMnO_4 ; 800 cc digestion flasks.
2.....	{ 1.450 1.410 1.412 }	1.424			{ Same as above except one and one-half hours' digestion.
	{ 1.450 1.480 1.470 }	1.467	{ 6.71 6.72 6.79 }	6.74	{ Same as above except one hour digestion.
3.....	{ 1.471 1.414 1.465 1.472 }	1.443	{ 6.71 6.71 }	6.71	{ Same as above except two and one-half hours' digestion.
		1.469			{ Same as above except three hours' digestion.
4.....	{ 1.41 1.42 1.40 }	1.41	{ 6.70 6.69 6.65 }	6.68	{ Gunning; 20 cc H_2SO_4 ; 10 grams K_2SO_4 ; digested four hours.
5.....	{ 1.38 1.40 }	1.39	{ 6.67 6.67 }	6.67	{ Gunning; with 0.1 gram copper sulphate; one hour digestion.
6.....	{ 1.43 1.43 }	1.43	{ 6.70 6.62 6.67 6.72 6.83 }	6.66	{ Mercuric oxid; colorless; green with KMnO_4 ; one and one-fourth hours' digestion.
				6.775	{ Same as above except three hours' digestion.
7.....	{ 1.51 1.48 }	1.495	{ 6.85 6.85 }	6.85	{ Mercury; colorless; green with KMnO_4 ; five hours' digestion; distilled in current of steam.
8.....	{ 1.45 1.43 1.47 1.47 1.47 }	1.44	{ 6.75 6.75 6.77 6.79 6.83 }	6.75	{ Mercury; three hours' digestion; no KMnO_4 .
		1.47		6.78	{ The same as the last, except KMnO_4 to permanent coloration.
		1.47		6.83	{ The same, except five hours' digestion and no KMnO_4 .
	{ 1.47 1.46 1.465 1.465 }	1.465	{ 6.83 6.83 6.84 6.82 }	6.83	{ Mercury; 10 grams K_2SO_4 ; three hours' digestion; no KMnO_4 .
		1.465		6.84	{ The same, except five hours' digestion.
		1.465		6.82	{ The same as the last, except KMnO_4 to permanent coloration.
9.....	{ 1.453 1.460 1.453 }	1.455			{ 12 grams K_2SO_4 ; 0.5 gram Hg; no KMnO_4 ; two and one-fourth hours' digestion.
			{ 6.873 6.873 6.852 6.860 6.839 6.846 6.851 }	6.866	{ The same, except three hours' digestion.
10.....	{ 1.442 1.442 1.435 1.435 1.456 1.456 1.449 1.449 }	1.439	{ 6.860 6.839 6.846 6.851 6.830 6.818 6.860 6.965 6.958 }	6.857	{ Official Kjeldahl; KMnO_4 used; one hour digestion.
		1.453		6.836	{ Kjeldahl-Gunning; Hg but no KMnO_4 ; two-thirds hour digestion.
				6.962	{ Same as last, except one hour digestion.

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
11.....	{ 1.50 1.44 }	1.47	{ 6.78 6.54 }	6.66	{ Hg; one and one-sixth hours' digestion; KMnO ₄ .
12.....	{ 1.49 1.43 }	1.46	{ 6.69 6.60 }	6.45	{ Same as above, except digested till colorless; time not given.
13.....	{ 1.43 1.46 1.40 1.46 1.46 }	1.43	{ 6.74 6.76 6.60 6.64 6.68 }	6.75	Same as above, but time not given.
14.....	{ 1.46 1.46 1.46 1.50 1.50 }	1.47	{ 6.60 6.64 6.68 }	6.64	Same as last.
15.....	{ 1.50 1.52 }	1.51	{ 6.80 6.80 6.80 6.84 }	6.81	{ Kjeldahl; one and one-fourth hours' digestion till colorless; KMnO ₄ .
	{ 1.54 1.56 }	1.55			{ Gunning; No KMnO ₄ ; digested two hours till colorless.
	{ 1.42 1.44 }	1.43	{ 6.82 6.80 }	6.81	{ Kjeldahl - Gunning; digested three- fourths of an hour till colorless; no KMnO ₄ .
			{ 6.74 6.70 6.88 6.88 6.92 6.92 6.86 6.86 }	6.72	{ Kjeldahl but no KMnO ₄ ; digested one and one-fourth hours till colorless.
				6.89	{ Kjeldahl with KMnO ₄ ; three hours' di- gestion.
16.....	{ 1.49 1.46 1.47 1.49 1.46 }	1.475 1.48 1.46	{ 6.82 6.86 6.84 6.88 }	6.84 6.86	{ Gunning; two and one-half hours' diges- tion. Kjeldahl; HgO and KMnO ₄ .
17.....	1.44	1.44	6.87	6.87	Hg; 10 grams K ₂ SO ₄ ; no permanganate; two hours' digestion; colorless. Same, except three hours' digestion. Same, except nearly colorless, faint yel- low; three and one-half hours' diges- tion.
			6.85	6.85	Same, except five hours' digestion.
18.....	{ 1.46 1.46 1.45 }	1.46			{ Dyer modification; i. e., Gunning with Hg and K ₂ S; no KMnO ₄ ; one and one- fourth hours' digestion.
			{ 6.83 6.76 6.79 6.85 6.74 6.78 6.79 }	6.79	Same, except two hours' digestion.
				6.795	Same, except one hour digestion.
			{ 6.72 6.74 6.63 }	6.785	{ Same, except three and one-half hours' digestion.
	{ 1.42 1.42 1.44 }	1.43		6.73	{ Kjeldahl method; digestion, two hours for corn; two and one-half for cotton seed.
				6.63	Same, except six and one-half hours' di- gestion. With Dyer modification solu- tions were quite colorless; those with Kjeldahl method showed faint color.
19.....	{ 1.43 1.43 1.40 1.40 1.42 }	1.43 1.41	{ 6.58 6.44 6.54 6.49 6.50 }	6.52 6.49	{ HgO; no KMnO ₄ ; digested two hours; corn was colorless; cotton seed pale straw color.
20.....					Same as last, except digestion fifty min- utes.
21.....	{ 1.416 1.429 }	1.423	{ 6.666 6.462 }	6.564	{ Kjeldahl; Hg; solutions nearly colorless with a slight yellowish tint in some cases; KMnO ₄ to permanent coloration.
22.....	{ 1.43 1.46 1.43 }	1.44	{ 6.37 6.34 }	6.355	{ Hg; digestion seventy minutes for corn and one hundred and ten minutes for cotton seed; hot solution of corn was colorless, of cotton seed very pale straw color; KMnO ₄ added to permanent col- oration.
23.....	{ 1.49 1.52 }	1.505	{ 6.49 6.49 }	6.49	{ Gunning; K ₂ SO ₄ and H ₂ SO ₄ only; six hours' digestion; solution practically colorless.

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
24.....	{ 1.40 1.42 1.42 1.40 1.42	{ 1.41	{ 6.65 6.64 6.64	{ 6.643	{Kjeldahl; Hg; no KMnO_4 ; digested one and one-half to two hours till colorless.
25.....	{ 1.45 1.45 1.44 1.44 1.44 1.46	{ 1.45 1.44 1.45	{ 6.82 6.82 6.87 6.84 6.68 6.70 6.75	{ 6.84 6.84 6.71	{Kjeldahl; HgO; digested three and one-half to four hours; colorless with corn, and straw color with cotton seed. Same as last, except Hg instead of HgO. {Same as the last, except KMnO_4 is omitted.
26.....	{ 1.389 1.412	{ 1.401	{ 6.663 6.705	{ 6.684	{Kjeldahl; five hours' digestion; faint yellow color; KMnO_4 to permanent green.
27.....	{ 1.368 1.389	{ 1.379	{ 6.621 6.636 6.678	{ 6.629	{Same as last, except four hours' digestion.
28.....	{ 1.372 1.428 1.442 1.400 1.414 1.414 1.428 1.428 1.428 1.40 1.41 1.39 1.36 1.40 1.38 1.39 1.39 1.37 1.38 1.39 1.37 1.40 1.42 1.41	{ 1.400 1.419 1.421 1.428	{ 6.734 6.692 6.650 6.692 6.692 6.748 6.650 6.720 6.748 6.748 6.776 6.68 6.68 6.72 6.68 6.70 6.72 6.66 6.74 6.78 6.70 6.74 6.70 6.74 6.76 6.80 6.74 6.74 6.70 6.70	{ 6.701 6.678 6.706 6.757	Kjeldahl, but no KMnO_4 and no CuSO_4 . {Kjeldahl and 0.2 gram CuSO_4 ; $5\text{H}_2\text{O}$; no KMnO_4 . Gunning, without CuSO_4 . {Gunning, with 0.2 gram CuSO_4 ; $5\text{H}_2\text{O}$; copper sulphate reduces time of digestion one-half.
29.....	{ 1.39 1.40 1.39 1.38 1.39 1.37 1.38 1.39 1.39 1.37 1.38 1.39 1.37 1.40 1.42 1.41	{ 1.39 1.39	{ 6.66 6.74 6.78 6.70 6.74 6.70 6.74 6.76 6.80 6.74 6.74 6.70 6.70	{ 6.71 6.735	{Kjeldahl; digested one and one-half hours; colorless; KMnO_4 to permanent green. {Same as above, except digested two and one-half hours.
30.....	{ 1.43 1.39	{ 1.41	{ 6.825 6.765 6.855 6.83 6.73	{ 6.815	{Colorless; KMnO_4 to permanent coloration.
31.....	{ 1.40 1.42 1.455 1.435	{ 1.425	{ 6.835 6.85 6.855	{ 6.80	Do.
32.....	{ 1.44 1.46 1.40	{ 1.444	{ 6.755 6.865	{ 6.823	Do.
33.....	{ 1.43 1.43 1.40	{ 1.41	{ 6.66 6.66	{ 6.66	Gunning; four hours' digestion.
	{ 1.43 1.43 1.43	{ 1.43	{ 6.68 6.68	{ 6.68	Kjeldahl; four hours' digestion.
34.....	{ 1.50 1.53 1.53	{ 1.52	{ 6.84 6.84 6.87	{ 6.85	{Kjeldahl modified for nitrates; five hours' digestion; not colorless; KMnO_4 to permanent coloration.
35.....	{ 1.48 1.48	{ 1.48	{ 6.90 6.88	{ 6.89	{Kjeldahl; Hg; one and one-half to one and three-fourths hours' digestion; colorless; KMnO_4 to permanent coloration.
36.....	{ 1.50 1.351 1.344 1.365	{ 1.50 1.353	{ 6.88 6.587 6.608 6.601	{ 6.88 6.599	Gunning, with 0.5 gram copper sulphate. 10 grams K_2SO_4 and 0.5 gram Hg; no KMnO_4 ; not strictly colorless; one and three-fourths hours' digestion.
37.....	{ 1.422 1.419 1.419	{ 1.420	{ 6.867 6.893 6.871	{ 6.877	{Kjeldahl; HgO; digestion two hours and fifty minutes; colorless; KMnO_4 added to permanent pink.

TABLE II.—Percentages of nitrogen found in corn meal, etc.—Continued.

Number of analyst.	Nitrogen.				Description of method.
	Corn meal.		Cotton-seed meal.		
	Per cent.	Per cent.	Per cent.	Per cent.	
38.....	{ 1.40 1.44 }	1.42	{ 6.79 6.81 6.81 6.90 6.85 6.87 }	{ 6.803 6.873 }	{ Plain Kjeldahl; Hg; pale yellow color; KMnO ₄ to permanent coloration; one and three-fourths' hours digestion. Kjeldahl modified for nitrates; Hg; KMnO ₄ as above.
39.....	{ 1.650 1.651 1.612 1.629 1.635 1.659 }	1.629	{ 7.146 7.201 7.229 7.114 7.179 7.121 }	{ 7.163 }	{ Kjeldahl; HgO; colorless; KMnO ₄ to permanent coloration; digestion, corn three hours, cotton seed four and one-half hours.
40.....	{ 1.44 1.39 1.41 1.43 1.44 1.42 }	1.42	{ 6.84 6.85 6.82 }	{ 6.837 }	{ Gunning; no KMnO ₄ ; two hours' digestion.
41.....	1.48	1.48	6.88	6.88	Kjeldahl; HgO; colorless; KMnO ₄ to permanent coloration; one and one-half hours' digestion.
42.....	1.45	1.45	6.85	6.85	Do.
43.....	1.48	1.48	6.84	6.84	Do.
44.....	{ 1.344 1.316 1.43 1.42 }	1.330	{ 6.356 6.342 6.71 6.78 }	{ 6.349 6.71 6.78 }	{ Gunning; straw color; no KMnO ₄ ; two hours' digestion. Hg; colorless; one and one-half to two hours' digestion; no KMnO ₄ ; no K ₂ S. Hg; colorless; KMnO ₄ to green color; no K ₂ S.
45.....	1.45	1.45	{ 6.69 6.76 6.74 6.75 }	{ 6.735 }	Same as last except K ₂ S was added.
46.....	{ 1.40 1.40 }	1.40	{ 6.60 6.64 }	{ 6.62 }	Same as last except KMnO ₄ was omitted. HgO; slightly yellow; KMnO ₄ to permanent coloration; two to three hours' digestion.
47.....	{ 1.50 1.50 1.39 1.43 1.42 1.44 1.44 1.44 1.43 1.40 1.44 1.43 1.41 1.43 1.44 1.46 }	1.50	{ 6.83 6.80 6.68 6.76 6.66 6.70 6.67 6.80 6.66 6.76 6.72 6.77 6.75 6.77 6.78 6.70 6.70 6.67 }	{ 6.815 6.70 6.71 6.75 6.747 6.767 6.69 6.66 6.725 }	{ HgO; two hours' digestion; colorless; KMnO ₄ to permanent coloration. Gunning; K ₂ SO ₄ and CuSO ₄ ; one and one-half hours' digestion; colorless; no KMnO ₄ . HgO; colorless; corn three hours and cotton seed five hours' digestion; KMnO ₄ to permanent coloration. Same as above except no KMnO ₄ . Gunning; three and one-half hours' digestion; colorless; no KMnO ₄ . Same as above with the addition of HgO; two hours' digestion. Hg; almost colorless; KMnO ₄ to permanent coloration; digestion three and one-fourth to four hours. Same as above except that after KMnO ₄ was added acid was boiled again until colorless. Hg; four and one-half hours' digestion; KMnO ₄ to permanent coloration.
50.....	{ 1.54 1.50 }	1.52	{ 6.70 6.70 6.67 }	{ 6.69 }	
51.....	{ 1.50 1.54 }	1.52	{ 6.67 6.65 }	{ 6.66 }	
52.....	{ 1.440 1.445 }	1.443	{ 6.73 6.72 }	{ 6.725 }	
Maximum.....		1.64		7.163	
Minimum.....		1.33		6.349	
Mean of all separate determinations.....		1.446		6.749	
Mean deviation.....		0.035		0.110	

(b) SODA LIME METHOD.

Number of analyst.	Corn meal.		Cotton-seed meal.	
	Results.	Average.	Results.	Average.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
21	{ 1.734 1.752 }	1.743	{ 6.934 6.855 }	6.895

The mean per cent of nitrogen determined is 1.446 for the corn meal and 6.749 for the cotton-seed meal. The various determinations range from 0.12 per cent below and 0.19 per cent above the mean for corn meal and for the cotton-seed meal 0.40 per cent below and 0.41 per cent above. If the mean of the several determinations be taken as the commercially correct figure, it appears that the average error is 0.035 per cent of nitrogen for corn meal and for cotton-seed meal 0.11 per cent. On the corn meal 41 out of 52 analysts, or 80 per cent, estimated within 0.05 per cent of the mean, and in the case of the cotton-seed meal 29 analysts, or 56 per cent, estimated within 0.10 per cent. This is a most satisfactory result, so far as these particular analysts are concerned, i. e., the 80 per cent and the 56 per cent, respectively. It seems probable that the limits just mentioned, 0.05 per cent for corn meal and 0.10 per cent for cotton-seed meal, above or below the accepted average, are the best that can be expected of a large number of analysts working in different laboratories and following a general description of a method. It would be desirable to make the limits of permissible error narrower, for mistakes are costly; but this represents the present state of accuracy and about one-fifth of our cooperating colleagues fail to attain even this limit in their corn meal determinations and over two-fifths that for cotton-seed meal.

The fact that a large number of analysts do agree closely is a most gratifying improvement over the experience of former years. It is doubtful if any further definition can be made to bring about a closer agreement. The only possible solution, in my opinion, is in concrete standards such as the samples sent out have now come to be.

The errors giving too low results may be explained by (1) a false weight (a most improbable source of error); (2) incomplete digestion, leaving some unreduced nitrogen; (3) excessive digestion, sometimes attended by the decomposition of ammonium sulphate; (4) mechanical loss from spurling; (5) incomplete distillation; (6) escape of unabsorbed ammonia in distilling, either through loose fittings or an unsubmerged outlet tube; (7) a false standard acid. Errors in excess are harder to explain. They may be due to (1) a false weight; (2) the incrustation of flasks with ammonium chlorid; (3) the carrying over of caustic alkali; (4) a false standard acid; (5) impure reagents; (6) with a glass condenser a possible solution of alkali.

A common fallacy in connection with this subject is that the differences in moisture contribute much to the variation in percentage of nitrogen. But a simple calculation will show that the extreme variations in moisture, as estimated, would cause a difference of 0.06 per cent nitrogen in the corn meal, and of 0.24 per cent in the cotton-seed meal. Furthermore, as it happens, the greater part of the analyses of both samples show that the moisture and nitrogen deviate from the mean in the same direction, i. e., both greater or both less; whereas if variation in moisture caused or contributed to the variation in nitrogen content the two quantities should vary in opposite directions. The only reasonable inference is that personal carelessness or inaptitude causes the widest deviations from the mean. After a consideration of many analyses, therefore, it would seem that 0.05 per cent of nitrogen in corn meal and 0.10 per cent in cotton-seed meal, are fair allowances for deviation. It is believed that the two samples submitted to the association have been analyzed accurately. The one can not be far from 1.45 per cent and the other not far from 6.75 per cent of nitrogen. They are hermetically sealed and nearly enough alike (as regards loss or gain of moisture) to serve as useful standards of comparison. The mean figures may not be absolutely free from error when the sulphuric acid method is used (the Kjeldahl method with its various amend-

ments and modifications), but as this method is followed almost exclusively in commercial work and investigations, they are at least conventionally correct.

Determinations by the soda lime method were made by one analyst, with results considerably higher than the other averages. But the data are so meager that no reliable conclusion can be drawn.

Four recommendations are made. The first one calls for from 20 to 30 cc of sulphuric acid instead of merely 20 cc, on the ground that there is danger of the flame striking the bare wall of the flask and decomposing some of the ammonium sulphate. The third one requires the condenser tube to dip below the surface of the standard acid on the ground that when there is much ammonia some may easily escape unless the tube is submerged. The second and the fourth permit the use of copper sulphate, in both the Kjeldahl method proper and in the Gunning modification of it. The claims made for the catalytic action of copper sulphate seem to be justified by experience, and there is reason to suppose that this reagent will do much to shorten the time of digestion. It is well nigh inconceivable that its presence can be in anyway hurtful, and there seem to be many analysts who are very desirous of using it and at the same time of conforming strictly to the official methods.

It is recommended that—

(1) Bulletin 107, p. 6, under (3) Determination, third line of first paragraph, be changed from "20 cc" to "20 cc-30 cc" (i. e., of sulphuric acid).

(2) Ibid., line 4, after "acid," add "From 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury, or in lieu of it."

(3) Ibid., p. 6, line 6 from bottom, after "standard acid," add: "During at least the first ten minutes of distillation the lower end of the condenser tube should dip beneath the surface of the standard acid."

(4) Ibid., p. 7, (b) Gunning Method, (3) Determination, line 4, after "sulphuric acid," add: "From 0.1 to 0.3 gram crystallized copper sulphate may also be added."

(5) Ibid., p. 7, (3) Determination, line 4, after "sulphuric acid," write: "Approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury may also be added, previously to the addition of the potassium sulphate, but if mercury be used potassium sulphid must be used, as in the Kjeldahl method, in the distillation."

A general discussion of the third recommendation made by the referee followed, showing a wide variation in practice and opinion regarding this detail of manipulation. The recommendation made was said to be in harmony with the practice in the Bureau of Chemistry, while Mr. Lipman stated that on a thousand determinations made by the New Jersey station without having the tube dip beneath the acid, almost perfect agreement was obtained. Mr. Patrick and Mr. Haskins spoke of the satisfactory results obtained by using a trap if the procedure recommended by the referee were not followed, and Doctor Wiley and Mr. Hopkins called attention to the bearing of temperature on the question. [It is to be noted that in the case of soils, fertilizers, and other low-grade materials, the variation introduced by this loss of ammonia is much less marked than when such materials as meat, cheese, and milk are under consideration. This recommendation was not adopted when presented to the association by Committee A (see page 129).]

REPORT ON THE SEPARATION OF MEAT PROTEIDS.

By F. C. Cook, *Associate Referee*.

Samples of commercial meat extract and the following letter of instructions were sent to the seven collaborators, who expressed a willingness to cooperate on meat proteids this year:

SEPTEMBER 4, 1907.

DEAR SIR: I am sending you, under separate cover, a sample of meat extract. The sample should be kept cool until ready for work, then opened and well mixed.

DIRECTIONS.

Total nitrogen. (1) Determine the total nitrogen in not over 0.5 gram sample.

Soluble nitrogen. (2) Dissolve 10 grams of the extract in water and make up to 500 cc. Filter on a plain filter paper, pouring back the first 10 or 20 cc. This is filtrate A. Determine the soluble nitrogen in 25 cc of filtrate A.

For the acidity determinations use two 10 cc portions of filtrate A. In one case use phenolphthalein as indicator, diluting the solution before titrating. In the second case employ delicate litmus paper, testing by taking a drop outside by means of a small capillary tube.

In determining the coagulable proteid take two 50 cc portions of filtrate A.

To No. 1 add 9 grams of sodium chlorid, heat to boiling, add 1 cc N/10 acetic acid and boil three minutes. Let stand on steam bath ten minutes, filter, and wash with 50 cc hot water.

Heat No. 2 to boiling and add 1 cc of N/10 acetic acid, boil three minutes, let stand on steam bath ten minutes, filter, and wash with 50 cc hot water.

In both cases determine the nitrogen on the filter paper. Use nitrogen-free filter paper.

The amido nitrogen is determined by placing 25 cc of filtrate A in a 100 cc flask. Add 15 grams of sodium chlorid and 25 cc of water. Shake and keep cool (15° C. or less) for two or more hours. Make a 24 per cent tannic-acid solution, filter and keep at same temperature. Add 30 cc of the 24 per cent tannic-acid solution to the flask, fill to mark with water, shake, and keep cool (15° C. or less) for twelve hours (usually overnight). Filter off 50 cc of the solution and determine nitrogen therein by adding a few drops of sulphuric acid and evaporating to dryness on the steam bath with help of vacuum if at hand. Add 25 or 30 cc of sulphuric acid but no potassium sulphate and proceed as in the Gunning method. It is necessary to run a blank, as the tannic acid often contains nitrogen.

The nitrogen figure minus the blank nitrogen figure multiplied by 2 equals amido nitrogen in the filtrate from 25 cc of the original sample (0.5 gram).

Kreatinin determination. Coagulate 20 cc of filtrate A, as in No. 2 under coagulable proteid, filter, and wash with hot water. Place the filtrate in a 500 cc flask, add 15 cc of saturated picric acid solution and 5 cc of 10 per cent sodium hydroxid, shake well. After standing 5 minutes dilute to mark with water and compare the color with an N/2 potassium dichromate solution in a Duboscq colorimeter, the scale being set at 8 mm; 81 divided by reading x on the other scale equals milligrams of kreatinin. This is a modification of Folin's method as applied to the estimation of kreatin and kreatinin in the urine.^a For the determination of kreatin use 15 cc of filtrate A, coagulate as above, filter, and wash. Place in an Erlenmeyer flask. Add 5 cc of N/2 hydrochloric acid and attach a reflux condenser, place on steam bath for 3½ hours, cool, transfer to a 500 cc flask, add 5 cc of N/2 sodium hydroxid and proceed as above, adding 15 cc picric acid, etc. This reading gives the total kreatinin. From the total kreatinin subtract the original kreatinin and multiply the difference by 1.16 to obtain the kreatin originally present.

Report results as follows:

Total nitrogen, per cent.

Soluble nitrogen, per cent.

Coagulable nitrogen, per cent.

Amido nitrogen, per cent.

Kreatinin, per cent.

Kreatin, per cent.

^a Zts. physiol. Chem., 1904, 41: 223; Amer. J. Physiol., 1905, 13: 48.

Acidity (1) using litmus cc N/10 sodium hydroxid per 100 grams.

(2) using phenolphthalein cc N/10 sodium hydroxid per 100 grams.

Coagulable nitrogen, using sodium chlorid, per cent.

Duplicate determinations should be made. Any criticisms and suggestions will be gladly received.

DISCUSSION OF RESULTS.

In the sample sent out there was but a small amount of insoluble proteid present, consequently no conclusion can be drawn under that head. The cooperative results as given in Table I show a wide variation for total nitrogen, and consequently for the different nitrogenous constituents, as the nitrogen is the basis of the determination.

TABLE I.—*Cooperative work on meat proteids.*

Analyst.	Nitrogen found as—					
	Total.	Soluble.	Insoluble by difference.	Coagulable.	Proteose and peptone.	Amido.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Grindley & Mitchell, Urbana, Ill.....	8.84	8.67	0.17	0.01	3.92	4.74
A. W. Dox, Storrs, Conn.....	9.23	9.22	.01	.01	4.13	5.18
Tottingham and Hart, Madison, Wis....	9.60	9.42	.18	.055	3.595	5.77
E. W. Rockwood, Iowa City, Iowa:						
Chemist 1.....	10.20	8.42	1.78	.76	3.77	3.89
Chemist 2.....	10.12	8.23	1.89	.80	4.20	3.23
F. C. Cook, Washington, D. C.....	9.30	9.21	.09	.185	5.095	3.93
P. Rudnick, Chicago, Ill.....	9.40	9.40	.00	.00	4.13	5.27
W. D. Richardson, Chicago, Ill.....	9.68	9.66	.02	.020	6.78	2.88
Lowenstein and Dunne, Chicago, Ill.....	9.20	8.93	.27	.000	3.30	5.63
H. C. Jackson, Albany, N. Y.....	9.71	9.56	.15	.072	3.14	6.35

Analyst.	Kreatin.	Kreatinin.	Acidity (N/10 alkali per 100 grams).		Coagulable proteid-nitrogen, using sodium chlorid.
			Phenolphthalein.	Litmus.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>cc.</i>	<i>cc.</i>	<i>Per cent.</i>
Grindley & Mitchell, Urbana, Ill.....	2.39	4.02	1242	-----	0.01
A. W. Dox, Storrs, Conn.....	-----	-----	1281	1255	.03
Tottingham and Hart, Madison, Wis.....	.348	7.90	1208	933	.102
E. W. Rockwood, Iowa City, Iowa:					
Chemist 1.....	.85	.80	1697	-----	.66
Chemist 2.....	.45	.85	1697	-----	.64
F. C. Cook, Washington, D. C.....	.371	4.50	1325	1025	.305
P. Rudnick, Chicago, Ill.....	1.38	3.24	1400	975	.000
W. D. Richardson, Chicago, Ill.....	2.41	3.21	1350	1000	.018
Lowenstein and Dunne, Chicago, Ill.....	.51	4.22	1450	1500	.000
H. C. Jackson, Albany, N. Y.....	-----	-----	1850	1238	.102

The tannin-salt method gave fairly satisfactory results, although there is still considerable variation in the results obtained by the different collaborators. The trouble often experienced by the foaming in the Kjeldahl process, due to the presence of the tannin, is eliminated by evaporating the solution, after adding 2 or 3 drops of sulphuric acid, almost to dryness, then adding about 30 cc of sulphuric acid and no potassium sulphate, and digesting as usual. The proteose and peptone figures reported were obtained by difference.

Cooperation was also obtained on the determination of coagulable proteids. There has been considerable discussion as to the use of sodium chlorid for this purpose, and the collaborators were instructed in one case to add 9 grams of sodium chlorid to 50 cc of the meat extract solution, while in the other case no sodium chlorid was to be added. As would be expected, practically all of the

results were higher when sodium chlorid was used, but as the amount of coagulable proteid present in the sample was small the results are not striking.

In Table II some results obtained by the referee are given, showing the influence of varying amounts of sodium chlorid on the coagulation of proteid in a commercial meat juice.

TABLE II.—*Coagulable proteid tests using varying amounts of sodium chlorid and 50 cc of the meat extract solution.*

Num- ber.	Sodium chlorid added.	Nitrogen precipi- tated.	Num- ber.	Sodium chlorid added.	Nitrogen precipi- tated.
	<i>Grams.</i>	<i>Grams.</i>		<i>Grams.</i>	<i>Grams.</i>
1	0	0.00842	6	6	0.01263
2	0	.00870	7	9	.01516
3	3	.01039	8	9	.01516
4	3	.01011	9	18	.01881
5	6	.01263	10	18	.01909

These figures show that the amount of nitrogen precipitated increases with the amount of sodium chlorid used, and Mr. Lowenstein, chemist for Morris & Co., Chicago, has obtained similar results for coagulable proteids on samples of meat extracts. It is evident, therefore, that to obtain correct figures for coagulable proteid nitrogen no sodium chlorid should be employed.

The two indicators, phenolphthalein and litmus, used in determining the acidity of meat products were compared, and the results obtained by the various chemists show that in practically all cases higher results were obtained where phenolphthalein was used. In a good many cases the difference is marked. The reason for this is that many of the organic acids have no action on litmus, but do act on phenolphthalein. The referee finds that phenolphthalein inevitably gives higher results than litmus, is easier to handle, and the end point is more accurate, but in certain dark-colored solutions of meat preparations litmus paper must be used.

In Table III the results of acidity tests on solutions of meat extract and on similar solutions after removing the insoluble proteid by filtering, and after coagulating and filtering, are given:

TABLE III.—*Acidity determinations.*

[Comparing the original solution, the solution after filtering, and the solution after coagulation and filtering, using two indicators.]

Num- ber.	Sample		Indicators (N/10 alkali).	
	Amount.	Description.	Litmus.	Phenolphthalein.
	<i>cc.</i>		<i>cc.</i>	<i>cc.</i>
1	25	Meat extract solution.....	4.20	7.575
2	25	Meat extract solution, filtered.....	2.55	7.350
3	25	Meat extract solution, coagulated and filtered.....	3.35	7.575
4	10	Meat extract solution.....	2.20	2.50
5	10	Meat extract solution, filtered.....	2.00	2.50
6	10	Meat extract solution, coagulated and filtered.....	1.80	2.95
7	20	Commercial beef juice, diluted.....	16.30	
8	20	Commercial beef juice, filtered.....	10.15	
9	20	Commercial beef juice, coagulated and filtered.....	8.50	

As the amount of insoluble proteid present in most of the samples was small, the results on this point are not conclusive, but the two latter sets of figures show lower results than those obtained by titrating the original solution, and in two cases the removal of the coagulable proteid before titration gives the lowest results. This is true only when litmus is used, the phenolphthalein

results showing but little variation among themselves. As the amount of proteid present in meat products affects the acid determination, a uniform procedure should be adopted. In the case of meat extracts, most of which contain little insoluble and coagulable proteid, the titration of the original solution is recommended.

The determination of kreatin and kreatinin was also investigated this year. According to the method suggested for kreatinin 20 cc of the coagulable proteid filtrate are used for the determination, but as this filtrate has previously been treated with 1 cc of N/10 acetic acid and boiled three minutes, the heat and acidity employed may have changed some kreatin to kreatinin, thereby giving too high results, consequently it is better to apply the kreatinin test to the original solution. This is satisfactory in the case of most meat extracts, as they contain little insoluble proteid. The effect of the presence of a considerable amount of proteid on the kreatinin test has not been determined.

The best method of applying to meat products, especially meat extracts, the kreatin test, including an estimation of both kreatin and kreatinin in the form of kreatinin, has not been evolved. There are several points which remain to be settled. Folin originally applied his method to 10 cc of urine. In the method on which cooperation was requested a much larger volume of the solution was used. Undoubtedly the volume has some influence on the results, and this point needs investigation. Hehner^a in a recent article claims that the addition of 15 cc of picric acid is not sufficient, and that, upon adding 25 or 30 cc, higher and more accurate results are obtained. This point was investigated, using a solution of pure kreatin and a meat extract, and the addition of 30 cc of picric acid gave but slightly higher results than when 15 cc were used.

The question of the amount of acid to be added for the conversion of kreatin into kreatinin, as well as the period of heating, are important factors. The referee has experimented with the cooperative method which is given in the letter of instruction, with the method as outlined by Grindley and Wood,^b and that used by Armour & Co., which is as follows:

Make the volume of the solution up to 25 cc, place in a 6-inch porcelain evaporating dish, add one-half its volume of normal hydrochloric acid, and evaporate nearly to dryness on the water bath. Add 25 cc of water and 12.5 cc of normal hydrochloric acid, and repeat the evaporation to dryness. Take up the residue in 25 cc of water, add 15 cc of picric acid and 5 cc of sodium hydroxid, let stand five minutes, and then rinse into a 500 cc volumetric flask.

This method gives good duplicates and is short and simple. With Grindley's method the duplicates were not so good nor the results so high, and the cooperative method gives lower results than either of the two others. It is evident that three and one-half hours' heating on the steam bath is not sufficient to convert all the kreatin to kreatinin. A method using 10 cc of normal hydrochloric acid and heating four hours in a boiling-water bath gives results that are fully as high as those obtained by Armour & Co. While none of the methods for kreatin and kreatinin are as yet entirely satisfactory, the value of these two determinations in analyzing meats and meat products has been generally recognized. Benedict and Myers^c have employed an autoclave for the conversion of kreatin to kreatinin. By this method fifteen minutes suffice for the complete conversion. The referee has this method under investigation and recommends that the study of an official method for kreatin and kreatinin be continued next year.

^a Pharm. J., 1907, 78: 683.

^b J. Biol. Chem., 1907, 3: 49.

^c Amer. J. Physiol., 1907, 18: 397.

One of the important features of this work is the criticism which it calls forth. The comments of the collaborators, whom the referee here takes occasion to thank for their helpful interest in the work, are as follows:

COMMENTS BY ANALYSTS.

H. S. Grindley, of Urbana, Ill., says: First, I do not think that the operation of determining the soluble nitrogen is of sufficient value to justify the additional labor.

Second, I think there is serious objection to the determination of kreatinin in the filtrate obtained after the coagulation of the solutions by heat and acetic acid. It is my opinion that by such an operation kreatin will be converted into kreatinin. Investigations made in this laboratory conclusively prove that the kreatin existing in water extracts of flesh are very readily, in part at least, converted into kreatinin even in the presence of the neutral acidity of the extract of flesh.

Third, the filtration of the solutions is very slow and tedious, and we have found that unless three or four filters are used for each solution there is an error in all determinations upon the filtrate due to the evaporation of the solutions during filtration.

Fourth, since the filtration of a water solution of meat extract is so difficult, it appears that, as far as possible, determinations should be made directly upon the original solution without filtration. Our work demonstrates that the presence of proteids, even in considerable quantity, does not interfere with the kreatin and kreatinin determinations.

Fifth, the acidity of ordinary extracts can be determined as well upon the solution of the extract before as after filtration. However, we have not found any meat extract which contains any considerable quantity of insoluble matter.

Sixth, the titration of the solutions of the extracts when using litmus as an indicator was very unsatisfactory. Men in this laboratory working independently have obtained such results when using litmus as to indicate clearly the unreliability of this indicator. For these reasons, no results of acidity of meat extracts when litmus is used as the indicator are reported.

Seventh, the tannin-salt method worked very nicely as to details of manipulation, and the duplicate results have been very satisfactory. On the other hand, it seems very unfortunate that a reagent which involves such a large correction factor must be employed. Although we have used Kahlbaum's very best tannin, it contained a large quantity of nitrogen.

Arthur Lowenstein, of Chicago, Ill., makes the following suggestions and criticisms regarding the method:

Total and soluble nitrogen: The methods for these determinations are quite satisfactory providing the sample is absolutely uniform, a difficult matter to accomplish with solid extracts containing 24 per cent moisture or less, and when only 0.5 gram of extract is employed an error is apt to be introduced.

Acidity: With the majority of extracts it is preferable to employ phenolphthalein as indicator. The reaction with litmus is not very sharp and with very highly colored extracts, such as are frequently found, the use of an outside indicator is necessary.

Coagulable nitrogen: The extract submitted contained practically no coagulable matter. However, in other samples the addition of sodium chlorid gave a higher result in every case.

Amido nitrogen: The method of evaporation is slow, and equally good results were obtained if 50 cc of the filtrate were used after precipitation with tannic

acid and digested immediately with sulphuric acid. The digestion with sulphuric acid only is extremely slow.

There seems to be quite a difference in the various tannic acids (c. p.) on the market. Some produce a very voluminous precipitate, others comparatively little. No account is taken in the method of the volume (bulk) of this precipitate. If it is considerable (as it might be in some extracts), it would introduce the same error that one finds in some of the present official methods.

Kreatinin: A criticism pertinent at this point, but also applicable to the methods as a whole, is they are not specific enough. I could readily observe in our own laboratory that the personal equation would be quite a factor. As this is the first year's work on this subject, just such points should be brought out. The method for kreatinin states, "take 20 cc of A, coagulate, filter, and wash." As the sample in question contains no coagulable proteid, we made the kreatinin determination both with and without dilution (washing), and found that the volume of the solution is quite a factor. Since the proposed method is a modification of Folin's method, in which he uses approximately 10 cc of urine, we thought that the more concentrated the solution the better would be the results obtained, and our results seem to confirm this view. Consequently we avoided transferring and washing as much as possible. Hehner states that 15 cc of picric were not sufficient for meat extracts, and also gives the amount of picric acid in solutions saturated at different temperatures, and this ought to be mentioned. Time, unfortunately, did not permit us to confirm Hehner's results, and in the work reported the quantities given in the method by the referee were used. I have heard from chemists who used 25 cc of picric acid, however, that they did not get results nearly so high as Hehner reports.

Kreatin: To determine whether the time of heating the solution on the steam bath was a factor, one determination was left on the steam bath for fifteen hours. The results checked very satisfactorily with the three-and-one-half-hour test.

The Grindley-Woods method was not tried because of lack of time, but we would suggest that after converting into kreatinin the sodium hydroxid and picric acid be added to the original Erbeumeyer flask and not transferred to the 500-cc flask until ready to dilute at the end of five minutes.

One of the principal objections to reporting these results was the age of the sample at the time of analysis. From practical experience it is known that extracts change with age, in fact, seem to improve in flavor in the case of solid or paste extracts, and we did not know whether the changes would be appreciable nor how much they would affect the content of meat proteids and bases and the acidity test.

Mr. Holmes C. Jackson, of Albany, N. Y., writes:

(1) As regards the acidity determination, it would seem to me that the 10 cc portions which were required to be used were a somewhat small fraction of the total, since in calculating the final figures (cc N/10 NaOH per 100 grams) it is necessary to multiply the titration figures by 500. Obviously very slight variations in the titration will give large variations in the final results. As stated last year, I am decidedly in favor of phenolphthalein as an indicator as compared with litmus, since, unless one makes his own paper, and neutral paper at that, I can not see how the necessary accuracy can be obtained.

(2) Concerning coagulable proteid, it is suggested that some mechanical feature in the method be introduced so that during the boiling and heating in the steam bath evaporation does not take place. Although not mentioned in the

method sent out, some such scheme was found to be absolutely necessary. I used a simple unjacketed reflux condenser.

(3) As regards the amido nitrogen, it seems to me that the results can not be absolutely correct, and the cooperative work shows that the results obtained by the different analysts are not comparable. There must be a variable factor introduced as the result of the presence of such a volume of precipitate. I therefore question again the accuracy of multiplying by 2 the results obtained from 50 cc of the filtrate from the original 100 cc of the mixture of filtrate and precipitate. In determining this point, it is suggested that the double dilution method used in the determination of lactose in milk be employed or some similar procedure which takes into account the volume of the precipitate.

The first sample sent me was lost after I had made the coagulable nitrogen analysis, and it gave a much higher result than the second sample sent. If the second sample was the same as the first and had been standing about during that time, is it not possible that a change had taken place in the character of the coagulable proteid by which it was transformed into noncoagulable? Might not this help to explain the great variations in the cooperative results for this factor?

Mr. Arthur W. Dox, of Storrs, Conn., says: "The precipitation of coagulable proteid seems to be incomplete. I would suggest that a greater concentration of acid be used and that the precipitate be washed with water that has been slightly acidulated. With sodium chlorid a higher figure is obtained, although the concentration is not sufficient to cause a precipitation of proteoses. If the latter method is adopted, the precipitate should be washed with a solution of sodium chlorid, so that none of it will be redissolved."

Mr. E. W. Rockwood, of Iowa City, Iowa, says: "The principal difficulties encountered were the indistinctness of the end reaction with litmus and in the colorimetric determination of the kreatin and the kreatinin. The latter was very probably due to lack of experience in judging these shades. The work was verified, and I can not explain the discrepancy in the nitrogen on any other basis than that it was not easy to sample accurately a substance with so high a nitrogen content. Both chemists have had experience in Kjeldahl determinations and worked independently."

Some analyses of commercial samples of meat extract by the cooperative methods are given in Table IV, including analyses by Mitchell and Grindley, of the University of Illinois, and W. D. Richardson, of Swift & Co., of Chicago.

TABLE IV.—*Analyses of commercial meat extracts by referee's methods.*

H. H. MITCHELL AND H. S. GRINDLEY.

[Each figure represents the average of three determinations.]

Number of sample.	Nitrogen.		Coagulable proteid precipitated by—		Amido nitrogen.	Kreatinin.	Kreatin.	Acidity determined by phenolphthalein (N/10 NaOH per 100 grams).
	Total.	Soluble.	NaCl and acetic acid.	Acetic acid.				
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>cc.</i>
2278	7.50	7.20	0.10	0.05	4.13	2.25	2.04	1,173.0
2306	9.01	9.02	.10	.03	5.82	5.32	1.60	1,194.0
2307	8.11	7.98	.03	.06	3.89	2.22	2.05	598.0
2308	6.62	6.42	.06	.07	3.27	1.19	.68	1,304.0
2309	9.11	7.96	.01	.01	5.74	3.69	1.28	990.0
^a 2330	8.84	8.67	.01	.01	4.74	4.02	2.39	1,242.0

^a Beef extract from the Bureau of Chemistry, U. S. Dept. Agr.

TABLE IV.—*Analyses of commercial meat extracts, etc.*—Continued.

W. D. RICHARDSON.

Sample.	Moisture.	Salt.	Ash.	Nitrogen.					Krea- tin.	Krea- tinin.	Acidity as indicated by (N/10 NaOH per 100 grams).	
				To- tal.	Solu- ble.	Coagulable.		Ami- do.			Lit- mus.	Phe- nol- phtha- lein.
						With salt.	With- out salt.					
"A," Extract of beef.....	<i>P. ct.</i> 19.54	<i>P. ct.</i> 7.04	<i>P. ct.</i> 23.15	<i>P. ct.</i> 8.56	<i>P. ct.</i> 7.90	<i>P. ct.</i> 0.283	<i>P. ct.</i> 0.176	<i>P. ct.</i> 3.65	<i>P. ct.</i> 0.75	<i>P. ct.</i> 1.89	cc. 900	cc. 1,150
"B," Beef extract..	20.6	15.43	29.97	7.24	7.18	.031	.088	4.35	.99	3.20	900	1,100
"C," Extract of beef.....	20.36	13.65	26.23	8.39	7.87	.019	{ .025 .03 }	3.59	.66	1.84	850	1,150
"D," Extract of beef.....												
"E," Beef extract..	23.62	11.09	26.87	8.11	7.59	.033	.036	4.32	1.13	1.40	850	1,050
"F," Extractum carnis.....	22.02	13.01	28.19	7.49	7.15	.026	.031	4.37	.39	2.92	1,000	1,150
"G," Beef fluid.....	21.07	3.32	18.29	9.98	9.36	.053	.159	4.96	2.07	3.11	1,300	1,500
	49.67	9.00	19.57	4.44	4.43	.024	.026	2.82	.01	1.60	675	925

RECOMMENDATIONS.

It is recommended—

(1) That the following method for the determination of the acidity of meat products be adopted as a provisional method:

Determine the acidity by titrating a dilute solution of the meat, or meat preparation, with N/10 alkali, using phenolphthalein as indicator. But if the color prevents the use of this indicator, use litmus paper with the understanding that the results are lower than when phenolphthalein is employed.

(2) That the modified tannin-salt method for the separation of proteoses and peptones from the simpler amido bodies be adopted as provisional. The method reads as follows:

Employ 50 cc of the filtrate from the determination of the coagulable proteids, transfer to a 100 cc graduated flask, add 15 grams of sodium chlorid, 10 cc of cold water, shake well and place in the cold at 12° C. (an ice box is a convenient place.) Prepare and filter a 24 per cent solution of pure tannin, and keep at the same temperature, 12° C., also a flask of distilled water. After the temperature of the contents of the flask has reached 12° C. add 30 cc of the tannin solution, shake thoroughly and fill to the mark with cold water, shake again and let stand at 12° C. for twelve hours (over night). Filter off 50 cc at 12° C. and determine the nitrogen therein as follows:

Transfer 50 cc of the tannin-salt filtrate to a Kjeldahl digestion flask and add a few drops of sulphuric acid. Place the flask in the steam bath, connect with the vacuum, and evaporate to dryness. In the digestion process about 30 cc of sulphuric acid are added, but no potassium sulphate. The remainder of the process is carried out in the usual manner.

A blank must be run simultaneously. The nitrogen in 50 cc of the filtrate above, minus the nitrogen in the 50 cc of the blank filtrate multiplied by 2, gives amido nitrogen. The nitrogen of the proteoses and peptones is determined by difference.

(3) That, inasmuch as the best modification of the Folin colorimetric method for the determination of kreatin and kreatinin in meat products has not been determined upon, this point be studied another year.

(4) That the referee for 1908 investigate the xanthin base method and a method for estimating organic phosphorus.

REPORT ON THE SEPARATION OF VEGETABLE PROTEIDS.

By H. SNYDER, *Associate Referee*.

The work on the separation of vegetable proteids has been confined to a study of the literature on the subject. No attempt has been made to correlate the literature, and only a general summary is given. No samples were distributed for the cooperative work, as previous investigations have shown that in the case of cereals, as in wheat, closely agreeing results can be secured when skilled analysts work on the same sample and follow a definite course of procedure.

There is such an extensive overlapping of the proteids as to solubility that it is doubtful whether accurate analytical methods can be devised for the absolute determination of any one proteid. The water-soluble, dilute salt-soluble, and alcoholic-soluble proteids can be determined with a reasonable degree of accuracy, but each solvent has a tendency to yield a mechanical mixture rather than an individual proteid. In the case of some of the cereals, however, a solvent may extract an individual proteid with a high degree of purity.

In the studies that are being made of the synthetic and ultimate composition of proteids, pure proteids are secured by special treatment not admissible in quantitative work. The object is to secure a pure proteid, and the mechanical losses due to the overlapping of the solvent can be disregarded.

At the present time it would seem best, in the determination of vegetable proteids, to designate the products as water-soluble, salt-soluble, or alcohol-soluble proteids, designating the proteid by the solvent rather than by the specific names suggesting individual proteids, except in special cases, as in wheat, where the alcohol-soluble proteid is mainly gliadin.

There is much need of an improved method for the determination of proteid and nonproteid nitrogen. While the determination of total nitrogen has reached a high degree of perfection, that for the determination of proteid nitrogen is defective. Stutzer's albuminoid nitrogen method does not make a sharp distinction between proteid and nonproteid nitrogen in many substances.

Instances occur where the copper proteid is soluble owing to secondary reactions. In the presence of a large amount of starch the filtration and washing of the precipitate often require hours, rendering the accuracy of the results questionable.

It would seem best first to direct attention to the improvement of the methods for the determination of proteid and nonproteid nitrogen rather than of individual proteids.

The analytical and synthetical work that is being done bids fair to give, in the near future, important results, and then it will be possible to devise rational methods for the separate determination of the various component proteids. To analyze accurately a complex compound, its composition must first be determined.

It is therefore recommended that the referee on vegetable proteids give special attention to the study of methods for the determination of proteid and nonproteid nitrogen.

The association adjourned to meet at 2.30 p. m.

THURSDAY—AFTERNOON SESSION.

On the suggestion of Doctor Frear, the recommendations in the president's address calling for changes in the constitution were referred to the committee on amendments to the constitution for recommendations.

In the absence of Mr. Woll, Mr. Bartlett, the associate referee, presented the report on dairy products.

REPORT ON DAIRY PRODUCTS.

By F. W. WOLL, *Referee*.

The referee was requested, by a vote of the association, to continue the study of the analysis of condensed milk with special reference to the determination of lactose, sucrose, and fat in the sweetened product.

The following explanatory letter, together with two carefully prepared samples of sweetened and unsweetened condensed milk, were sent to 22 different stations where chemists had signified their willingness to cooperate in the work outlined by the referee.

MADISON, WIS., *March 28, 1907.*

DEAR SIR: As stated in my circular letter of the 12th instant, the cooperative work on dairy products, Association of Official Agricultural Chemists, for this year will be along two lines:

I. *Analysis of condensed milk*, with special reference to the determination of sucrose and lactose in the sweetened product.

The provisional methods for analysis of condensed milk adopted tentatively at the last convention of the association are as follows:

Preparation of sample: Mix thoroughly by transferring the contents of a can to a large evaporating dish and stirring with a pestle. Weigh 40 grams into a 100 cc flask, transfer thereto by washing, and make up to the mark with water.

Total solids: Dilute a measured portion of the above 40 per cent solution with an equal amount of water, use 5 cc of the diluted mixture, and proceed as in the case of milk analysis.

Ash: Ignite the residue from the total solids, cool, and weigh.

Protein: Determine nitrogen by the Kjeldahl or Gunning method in 5 cc of the 40 per cent solution and multiply it by 6.25.

Lactose: Dilute 5 cc of the 40 per cent solution to about 40 cc and add 0.6 cc of Fehling's copper solution; nearly neutralize with sodium hydrate solution and make up to 100 cc. Filter through a dry filter and determine the lactose in an aliquot by the gravimetric and polariscope methods.

Sucrose: Determine (a) gravimetrically and (b) by difference, by deducting the milk solids (lactose, protein, fat, ash) from the total solids.

Fat: Determine by (a) double-extraction method (Circular 32, page 6, Bureau of Chemistry, U. S. Department of Agriculture) and (b) Babcock centrifugal method, using either Leach's modification (Food Inspection and Analysis, page 49) or Farrington's (Wisconsin Station Report 17, page 86).

II. *The Gottlieb method for the determination of fat in milk.*

The directions for the Gottlieb method are as follows (Landw. Vers. Sta., 1892, 40: 1-27):

Ten cc of milk are measured into a glass cylinder $\frac{3}{4}$ inch in diameter and about 14 inches long (see Land. Vers. Sta., 40: 6; a 100 cc burette or a eudiometer tube will do); 1 cc concentrated ammonia is added and mixed thoroughly with the milk. The following chemicals are next added in the order given: 10 cc of 92 per cent alcohol, 25 cc of washed ether, and 25 cc petroleum ether (boiling point below 80° C.), the cylinder being closed with a moistened cork stopper and the contents shaken several times after the addition of each. The cylinder is then left standing for six hours or more. The clear fat solution is next pipetted off into a small weighed flask by means of a siphon drawn to a fine point (see Fig. 6, loc. cit.), which is lowered into the fat solution to within 0.5 cm of the turbid bottom layer. After evaporating the ether solution in a hood, the flasks are dried in a steam oven for two to three hours and weighed. This method is applicable to new milk, skim milk, buttermilk, whey, cream, cheese, condensed milk, and milk powder, but has been found of special value for determining fat in skim milk, buttermilk, cheese, and condensed milk. In the case of products high in fat a second treatment with 10 cc each of ether and petroleum ether is advisable in order to recover the last trace of fat.

Chemists are asked to make comparative fat determinations in a number of samples of skim milk and buttermilk by this and the official method of fat determination in milk. * * *

The referee is indebted to the Borden Condensed Milk Company, New York City, N. Y., who furnished the samples of milk, together with what they believe to be "fair average analyses" of the two products.

TABLE I.—Results as reported by Borden Condensed Milk Company.

Name and number of milk.	Cane sugar.	Milk sugar.	Water.	Protein.	Fat.	Ash.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Sweetened, No. 9.....	43.86	12.08	25.47	8.06	8.75	1.78
Unsweetened, No. 123.....		12.24	70.81	6.97	8.20	1.36

Eight reports of the work done on these samples were received. The results of the analyses given in the following pages have been arranged for the sake of convenience under three headings: (1) Solids, protein, and ash in condensed milk. (2) Fat in condensed milk. (3) Sugar in condensed milk.

SOLIDS, PROTEIN, AND ASH IN CONDENSED MILK.

TABLE II.—Solids, protein, and ash in sample No. 9, sweetened condensed milk.

Analyst.	Solids.	Protein.	Ash.	Analyst.	Solids.	Protein.	Ash.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. M. Barnhart, Illinois..	74.32	7.59	1.46	G. E. Patrick and M. Boyle, Washington, D. C.	73.95	a 8.08	1.67
James M. Doran, Washington, D. C.	74.83	8.38	1.50	P. H. Smith and E. B. Holland, Massachusetts.	72.75	7.77	1.71
J. A. Hummel, Minnesota.	75.33	7.49	1.77	A. C. Whittier, Maine...	73.34	7.53	1.66
Geo. A. Olson, Wisconsin.	72.02	7.13	1.47				

TABLE III.—Solids, protein, and ash in sample No. 123, unsweetened condensed milk.

Analyst.	Solids.	Protein.	Ash.	Analyst.	Solids.	Protein.	Ash.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. M. Barnhart, Illinois..	29.55	7.32	1.34	G. E. Patrick and M. Boyle, Washington, D. C.	29.55	a 7.69	1.75
James M. Doran, Washington, D. C.	29.81	8.44	1.48	P. H. Smith and E. B. Holland, Massachusetts.	29.22	7.49	1.71
J. A. Hummel, Minnesota.	30.04	7.04	1.79	A. C. Whittier, Maine...	28.58	7.50	1.87
Geo. A. Olson, Wisconsin.	28.33	7.50	2.17				

a NX6.38.

REMARKS BY ANALYSTS.

G. E. Patrick: The solids obtained by drying a milk of any kind will vary from 0.1 to 0.2 per cent. according to the method of drying, i. e., whether dried rapidly or slowly, at high or low temperature. In the work here reported samples were dried first on the steam bath and then in boiling-water oven, at atmospheric pressure.

P. H. Smith and E. B. Holland: Moisture: Dried 15 cc of a 20 per cent solution on quartz sand in a flat-bottomed dish at a low temperature until bulk of

the water was expelled, then completed at 100°C. Ash: Evaporated to dryness 25 cc in a platinum dish with 5 cc of concentrated nitric acid and burned to white ash. Protein: Employed the Kjeldahl-Gunning method, using 5 cc of solution.

James M. Doran: Solids: Determined according to the provisional method of the association, evaporating first over steam bath and later drying in water oven to constant weight. Protein: Determined nitrogen in aliquot of diluted milk (corresponding to about two grains of the original sample) by the Gunning method.

Geo. A. Olson: Solids: Determined in the Babcock asbestos tube until constant in weight. Protein: Determined by the straight Kjeldahl method. Ash: Milk first dried in steam oven and finally incinerated in muffle furnace.

DISCUSSION OF RESULTS.

The results for protein and ash on the whole are fairly concordant. This, however, can not be said of those for solids. The difference in solids alone—3 per cent—is sufficient to throw the analyses of the various ingredients out of balance.

FAT IN CONDENSED MILK.

TABLE IV.—*Fat in sample No. 9, sweetened condensed milk.*

Analyst.	Asbestos method.			Babcock test.		Gottlieb method.
	First extraction.	Second extraction.	Total.	Leach.	Farrington.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....			7.78	8.1		
James M. Doran, Washington, D. C.....						9.24
J. A. Hummel, Minnesota.....				7.5		
Geo. A. Olson, Wisconsin.....	3.10	4.56	7.66	8.05	7.75	7.87
G. E. Patrick and M. Boyle, Washington, D. C.....	8.44	0.08	^a 8.52			^b 8.61
P. H. Smith and E. B. Holland, Massachusetts.....	5.38	2.14	^c 7.52			
A. C. Whittier, Maine.....			7.90	8.1		8.15

^a Adams paper coil method, 20 per cent solution, first extraction, 8.44; second, 0.08.

^b First extraction, 8.22; second, 0.31; third, 0.08.

^c Modified method, 7.65.

TABLE V.—*Fat in sample No. 123, unsweetened condensed milk.*

Analyst.	Asbestos method.			Babcock test.		Gottlieb method.
	First extraction.	Second extraction.	Total.	Leach.	Straight.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....			8.02	8.4		
James M. Doran, Washington, D. C.....						7.96
J. A. Hummel, Minnesota.....					7.20	
Geo. A. Olson, Wisconsin.....	8.10	0.37	8.47	8.1	8.20	8.41
G. E. Patrick and M. Boyle, Washington, D. C.....	8.11	.01	^a 8.12			^b 8.49
P. H. Smith and E. B. Holland, Massachusetts.....	6.47	.74	^c 7.21			
A. C. Whittier, Maine.....			7.91	8.1		8.32

^a Adams paper coil, 40 per cent solution, first extraction, 8.11; second, 0.01.

^b First extraction, 8.47; second, 0.02.

^c Modified method, 7.40.

REMARKS BY ANALYSTS.

G. E. Patrick: For comparison, fat was determined by the Adams method on a 40 per cent solution of the sweetened condensed milk, giving a result—the mean of triplicates—about 0.3 per cent lower than by the same method on a 20 per cent solution, as given above.

In the Gottlieb method for fat the Rohrig tube, mentioned in last year's report, has been used continuously with the greatest satisfaction. We consider the Gottlieb method the best known for the determination of fat in milks.

Geo. A. Olson: Adhered to methods as described in circular letter.

P. H. Smith and Edw. B. Holland: Method (A). Extracted the pulverized solids with dry ethyl ether in a continuous extractor before and after washing to remove sugars. Method (B). Washed solids with water to remove sugars and extracted with ethyl ether. Results higher, with a considerable saving of time and work.

James M. Doran: One to one and a half grams of the original sample was used for the Gottlieb method and allowed to stand over night before siphoning. The Adams-Soxhlet method gave low results after three days' extraction. It appeared to be unsatisfactory owing to the length of time required to obtain results.

DISCUSSION OF RESULTS.

As stated last year, the double-extraction method increased the per cent of fat in both sweetened and unsweetened milk. The Gottlieb method generally gives higher results than any one of the other methods employed. The lack of agreement in the different methods of estimating the fat may be, in part, due to the variations in the samples.

SUGAR IN CONDENSED MILK.

TABLE VI.—*Sugar in sample No. 9, sweetened condensed milk.*

Analyst.	Method.	Gravimetric.		Polariscope.	
		Lactose.	Sucrose.	Lactose.	Sucrose.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....	Walker.....	<i>a</i> 12.61			
A. H. Bryan, Washington, D. C.....	Soxhlet.....	<i>a</i> 12.18	<i>a</i> 42.49		
Do.....	Walker.....	<i>a</i> 12.71			
Do.....	Walker.....	<i>b</i> 12.09	<i>b</i> 42.56		
Do.....	Soxhlet.....	<i>b</i> 12.60			
Do.....	Walker.....	<i>c</i> 12.12	<i>c</i> 42.50		
Do.....	Soxhlet.....	<i>c</i> 12.63			
James M. Doran, Washington, D. C.....	Dubois.....	<i>a</i> 12.92	<i>a</i> 42.56		
A. Given, Washington, D. C.....	Walker.....	<i>a</i> 12.68	<i>a</i> 43.56		
Do.....	Soxhlet.....	<i>a</i> 12.96			
Geo. A. Olson, Wisconsin.....	Allihn.....	<i>b</i> 12.37	<i>b</i> 43.58		<i>d</i> 42.20
G. E. Patrick and M. Boyle, Washington, D. C.....	Soxhlet.....	<i>c</i> 11.58	<i>c</i> 43.14		43.51
Do.....	Walker.....	<i>c</i> 11.62			
P. H. Smith and E. B. Holland, Massachusetts.....	Defren O'Sullivan	<i>c</i> 11.97	40.81		
A. C. Whittier, Maine.....		12.65	42.75		41.30
M. H. Wiley, Washington, D. C.....	Allihn.....	<i>a</i> 12.80	<i>a</i> 43.67		

a Weighed as Cu_2O .

b Weighed as CuO .

c Low zinc acetate method.

d In another sample 42.72 per cent sucrose was obtained.

e Munson and Walker method.

TABLE VII.—*Sugar in sample No. 123, unsweetened milk.*

Analyst.	Method.	Gravimetric.		Polariscope.
		Lactose.	Sucrose.	Lactose.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. M. Barnhart, Illinois.....		12.17	None.	
A. H. Bryan, Washington, D. C.....	Walker.....	<i>a</i> 11.38		
Do.....	Soxhlet.....	<i>a</i> 11.51		
Do.....	Walker.....	<i>b</i> 11.20		
Do.....	Soxhlet.....	<i>b</i> 11.38		
Do.....	Walker.....	<i>c</i> 11.35		
Do.....	Soxhlet.....	<i>c</i> 11.50		
James M. Doran, Washington, D. C.....	Dubois.....	<i>a</i> 11.86		
A. Given, Washington, D. C.....	Walker.....	<i>a</i> 11.56		
Do.....	Soxhlet.....	<i>a</i> 11.48		
George A. Olson, Wisconsin.....	Allihn.....	<i>b</i> 10.90	None.	10.86
G. E. Patrick and M. Boyle, Washington, D. C.....	Soxhlet.....	<i>c</i> 11.48		11.12
Do.....	Walker.....	<i>c</i> 11.33		
P. H. Smith and E. B. Holland, Massachusetts.....	Defren O'Sullivan.....	<i>c</i> 11.15	0.67	
A. C. Whittier, Maine.....		9.79	None.	
M. H. Wiley, Washington, D. C.....	Allihn.....	<i>a</i> 11.56		

a Weighed as Cu₂O.*b* Weighed as CuO.*c* Low zinc acetate method.

REMARKS BY ANALYSTS.

P. H. Smith and E. B. Holland: Inverted with one-tenth its volume of concentrated hydrochloric acid, neutralized with sodium hydrate solution and proceeded as under reducing sugar. Calculations after deducting for the lactose by association tables, pages 35 and 37, Bulletin 46, Revised edition. Dextrose to sucrose by factor 0.95.

James M. Doran: Trials with some of the well-known methods have proved unsatisfactory. With proper conditions, however, in the inversion and temperature it is thought that both sucrose and lactose can be determined accurately.

G. E. Patrick: Lactose corrected for error caused by cane sugar.

C. A. Browne: You will note a variation in the lactose in sample No. 9 by the different methods, which I attribute to the varying degree of inversion of sucrose produced by the different methods of procedure. The Walker method requires only two minutes instead of six as by the Soxhlet, and uses much less alkali than the Allihn with decidedly less inversion in each case. In sample No. 123, where no sucrose was present, the results by the three methods are in very close agreement.

The error due to the inversion of sucrose in the gravimetric determination of sugars in sweetened milks seems to be as pronounced as in the analysis of sugar cane or beet products. We had no time to determine what correction should be made for this, but the experiments performed upon mixtures of lactose and sucrose (see following tables) indicate that an error of at least 0.50 per cent is involved in the case of the determinations by the Allihn method. Weighing the cuprous oxid gave accurate results with all samples, there being no contamination of the precipitate with organic or with mineral matter, as is often the case with molasses.

Effect of inversion of sucrose on the determination of lactose (M. H. Wiley).

Amount taken.		Amount found.
Lactose.	Sucrose.	Lactose.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
2.00		1.97
2.00	10.00	2.75
4.00		3.97
4.00	10.00	4.72
8.00		8.03
8.00	10.00	8.92
12.00		12.17
12.00	10.00	12.84

The Allihn method was used in all determinations, the dextrose being converted to lactose by the formula $\frac{D}{0.675} = \text{lactose}$. The lactose used had one-half molecule of water of crystallization.

Analysis of an artificial mixture of sucrose and lactose as found in condensed milk.

[M. H. Wiley and C. A. Browne.]

Amount taken.		Amount found.		
Lactose (one-half molecule of water).	Sucrose.	Allihn's method.		Clerget's method.
		Lactose.	Sucrose.	Sucrose.
<i>Per cent.</i> 12.00	<i>Per cent.</i> 42.00	<i>Per cent.</i> 12.60	<i>Per cent.</i> 41.47	<i>Per cent.</i> 42.14

Per cent sugar after inversion as dextrose equals 50.44; per cent sugar before inversion as dextrose equals 8.72; per cent reducing sugars as dextrose from sucrose equals 41.72.

Therefore $\frac{8.72}{0.692} = 12.60$ per cent lactose (one-half molecule of water).

$41.72 \times 0.993 = 41.47$ per cent sucrose.

DISCUSSION OF RESULTS.

The determination of sugars in condensed milk as reported this year indicates a very promising progress. The results as a whole show a fair agreement when one considers the variation in the total solids, which alone is sufficient to cause differences that would exceed those shown in the data reported. The determinations of sugars in unsweetened milk appear to be more uniform than those in sweetened milk. Polariscopic determinations for sucrose are generally lower than those obtained by gravimetric methods and may be attributed to the inversion of a part of the sucrose. The effect of inversion on the determination of lactose, brought about by the addition of sucrose, is interesting, and in consequence a higher lactose reading would be expected. It is evident that in future work along this line it will be necessary to determine the factor for correction in milk sweetened by sucrose.

RECOMMENDATION.

It is recommended—

(1) That the study of methods of analysis for condensed milk be continued and that special attention be given to the following methods for determining the fat and sugar content of condensed milk.

(a) Double extraction method for fat as described in the proceedings of the association for 1906.^a

(b) Gottlieb method as described in the proceedings of the association for 1906.^a

(c) Walker method for the gravimetric determination of sugar (J. Amer. Chem. Soc., 1907, 29: 541).

(d) Allihn method for the gravimetric determination of sugar.

(e) Polariscopic method for comparison with the gravimetric method for the determination of sugar.

G. E. PATRICK. In our laboratory we always correct the lactose result for the error caused by the presence of sucrose in analyzing sweetened condensed milks. We have not yet made a table of corrections, but have thus far determined the error in each sample by making a water solution of the two sugars corresponding to the results found, and in this solution—and a similar one of lactose without sucrose—finding the amount of the error in milligrams of copper oxid, which we then deduct from the copper oxid obtained in the analysis. In our experience thus far the correction has been from 12 to 15 milligrams by the Soxhlet method, and 8 to 12 milligrams by the Walker method on the charges prescribed by the official method for sweetened condensed milk.

P. H. WALKER. I have made a few determinations with mixtures of sucrose and lactose which would indicate that the correction needed would be very small. The lactose used, however, was the same as that employed in making up the tables published in the Journal of the American Chemical Society and had been standing for about eighteen months. Not sufficient work has been done to make up tables for this correction.

TWO METHODS IN CHEESE ANALYSIS.

By G. E. PATRICK.

The two following methods for the determination of moisture and the separation of fat in cheese analysis have been used with great satisfaction for several years in the Dairy Laboratory of the Bureau of Chemistry. They are presented to the association with the suggestion that it might be wise to include them in the methods, as in the writer's opinion they are far superior to the procedures there outlined.

1. WATER DETERMINATION.

Sift a quantity of asbestos by rubbing it through a wire screen—a window screen free from rust serves the purpose well. (Iron rust in the asbestos works mischief in the subsequent ash determination.) In a flat-bottomed platinum dish (aluminum may be used if the ash is not to be determined) of 6 to 7 cm diameter place about 2 grams (a little more rather than less) of the sifted

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

asbestos, pressing it down well over the bottom, then lay in the dish a thick glass rod cut off square at one end, to serve as a pestle. Ignite and weigh, then weigh into the dish about 5 grams of the cheese sample (prepared as usual) and with the glass rod rub and press the cheese and asbestos together most intimately, until all cheese particles have disappeared and the mass is homogeneous; finally, loosen the mass up into as fluffy a condition as possible, and place in the drying oven. Dry until loss of weight ceases, each drying to be for only an hour or an hour and a half. Usually two or three dryings suffice.

2. SEPARATION OF THE FAT FOR EXAMINATION.

Rub up thoroughly in a mortar 75 grams of the cheese with 90 to 100 cc of ether, throw the mass upon a cloth filter, press out the ether-fat solution as much as possible, return the mass to the mortar and repeat the treatment twice again (the second time less ether will suffice); filter the ether-fat solution through paper, recover the ether by distillation, filter the fat if it is not clear, then dry on the steam bath and finally in the oven until the ether is expelled, weighing every hour in order to guard against oxidation.

Petroleum ether boiling below 40° C. may be used, but has the fault of being expelled rather slowly from the fat. If only the neutral fat, free from uncombined fatty acids, is desired, add to the original extract two or three drops of phenolphthalein solution and then soda solution dropwise, with constant shaking, until the aqueous portion remains pink: then proceed as already described.

REPORT OF COMMITTEE C ON RECOMMENDATIONS OF REFEREES.

By L. M. TOLMAN, *Chairman*.

(1) MEAT PROTEIDS.

It is recommended that—

(1) The modified tannin-salt method for the separation of proteoses and peptones from the simpler amido bodies be adopted as a provisional method. (For statement of method, see referee's report, p. 51.)

The committee recommended that this method be further studied for another year before its adoption, and this motion was passed.

(2) That the following method for the determination of the acidity of meat products be adopted as a provisional method:

Determine the acidity by titrating a dilute solution of the meat, or meat preparation, with N/10 alkali, using phenolphthalein as indicator. But if the color prevents the use of this indicator, use litmus paper, with the understanding that the results are lower than when phenolphthalein is employed.

Adopted.

(3) That inasmuch as the best modification of the Folin colorimetric method for the determination of kreatin and kreatinin in meat products has not been determined, this point be studied another year.

Adopted.

(4) That the referee for 1908 investigate methods for the estimation of organic phosphorus and of xanthin bases.

Adopted.

(2) FLAVORING EXTRACTS.

It is recommended—

(1) That methods for the detection of caramel and other coloring matter in vanilla extracts be studied.

Adopted.

(2) That the quantity and quality of soluble matter in vanilla beans obtained in the preparation of standard vanilla extract be determined by the referee in 1908.

Adopted.

(3) DETERMINATION OF MOISTURE IN FOODS.

It is recommended that the work on this subject be continued another year.

Adopted.

(4) COLORS.

It is recommended that further work be done looking to a more comprehensive examination of the so-called "vegetable colors," some of which would seem to be no more properly classed as vegetable colors than are the anilin dyes.

Adopted.

(5) VEGETABLE PROTEIDS.

It is recommended that special attention be given to the study of methods for the determination of proteid and nonproteid nitrogen.

Adopted.

(6) COCOA AND COCOA PRODUCTS.

It is recommended that the methods submitted by the referee^a be adopted as provisional until a fuller report on the subject can be made, except that for section 13 on sugar the method proposed by Dubois for the determination of sugar in chocolate be substituted.

Adopted.

REPORT OF THE COMMITTEE ON THE REVISION OF METHODS.

J. K. HAYWOOD, *Chairman*.

The following report was presented by Mr. L. M. Tolman, acting for the chairman:

In accordance with the recommendations made in the report of the committee on revision of methods in 1906, and the suggestions of the secretary of the association made at that time, the revised methods were printed as Bulletin No. 107 of the Bureau of Chemistry and forwarded to the members of the association for criticism about three weeks before the 1907 meeting. Copies of this bulletin were sent to the following lists: Officers and referees of the association; directors and chemists of State agricultural experiment stations; superior officers of State boards of health, and chiefs of laboratories in the Bureau of Chemistry, Department of Agriculture.

The following letter was sent with each copy:

SEPTEMBER 12, 1907.

DEAR SIR: Inclosed you will find a copy of the revised methods of analysis of the Association of Official Agricultural Chemists recommended last year by the committee on revision and provisionally adopted by the association. Final action upon these revised methods will be taken this year.

You are respectfully requested to look these methods over at once, especially those with which you are most familiar. If you have any criticism to make of the changes incorporated, or can point out any errors, please report same to J. K. Haywood, Bureau of Chemistry, U. S. Department of Agriculture, Washington, D. C., chairman of revision committee, so that the letter will reach him before October 5, if possible, certainly not later than October 7. All criticisms will be considered by the committee at a meeting to be held in Washington on Octo-

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 107 Revised, p. 254.

ber 5 to 8, and recommendations made to the association at the Jamestown meeting on October 9 to 12, in regard to any further changes or additions, before the revised methods are officially adopted.

Respectfully,

H. W. WILEY,
Chief, Bureau of Chemistry,
Secretary, Association Official Agricultural Chemists.

Twenty-five chemists sent criticisms of the revised methods. These criticisms were carefully considered by this committee and, in the main, were adopted, the majority being simply corrections of obvious errors in spelling, punctuation, printing, etc. Some chemists suggested material changes in official methods, which were not adopted, as the committee did not feel that it had the power to make such far-reaching modifications. When changes were suggested to make the statement of the method more clear, but not to change the method itself, these were adopted.

The committee would respectfully recommend that the methods as now published in Bulletin 107 and further corrected, as indicated above, be adopted as the official and provisional methods of the Association of Official Agricultural Chemists.

(Signed)

J. K. HAYWOOD, *Chairman.*
 J. P. STREET,
 A. L. WINTON,
 F. P. VEITCH,
 L. M. TOLMAN,
 J. H. PETTIT,
 F. W. WOLL (absent).

Upon a motion being made to adopt the report, a discussion ensued as to the extent of the authority of the committee to change the methods by dropping obsolete procedures and otherwise bringing the methods up to date and the several sections into harmony with each other, especially when changes in official methods were involved.

The original motion was amended by a motion to continue the committee with authority to make such further changes of this nature as might be deemed necessary, said changes to be approved by the executive committee and inserted in the final revision. This motion was passed, and the original motion as amended was then carried, adopting the report of the committee and making the revision official.

A vote of thanks was passed by the association in appreciation of the work of the committee on revision.

REPORT ON FOODS AND FEEDING STUFFS.

By J. K. HAYWOOD, *Referee.*

On account of a great pressure of official work it was impossible for the referee to send out samples for examination; however, some comparative work was done by two chemists (Messrs. Goodrich and Houghton) of the Miscellaneous Laboratory of the Bureau of Chemistry, working entirely independently of each other,

The method of determining methyl pentosans, originated by Ellett and Tollens,^a and reported, in a modified form, by the referee last year, was first considered. Varying quantities of gum tragacanth, which is known to contain some methyl pentosans, were subjected to analysis by the following method:

METHOD FOR DETERMINING PENTOSANS AND METHYL PENTOSANS.

Proceed as in the determination of pentosans by the provisional method (Bulletin 107, page 54) until the phloroglucid precipitate has been dried for four hours and weighed. Place the Gooch crucible containing this precipitate in a 100 cc beaker and pour into the Gooch 30 cc of 95 per cent alcohol heated to 60° C. Place the beaker for ten minutes in a water bath heated to 60° C. Remove the beaker and Gooch and draw off from the Gooch with a suction pump all alcohol remaining therein. Repeat this alternate extraction and sucking dry of the precipitate from three to five times, according to the color of the filtrate obtained. After the final extraction place the Gooch crucible in a water oven and dry four hours, making the final weighing in a closely stoppered glass weighing bottle, as described in the provisional method for pentosans.

The difference in weight between the furfural-phloroglucid plus methyl-furfural-phloroglucid first obtained and the furfural-phloroglucid remaining after extraction with alcohol, minus 0.0037, represents the amount of methyl-furfural-phloroglucid present, from which the original pentose (as rhamnose) can be calculated by the following formula:

$$\text{Rhamnose} = (\text{Ph}) (165) - (\text{Ph})^2 (1.84) + 0.010.$$

in which "Ph" equals the weight of methyl-furfural-phloroglucid; rhamnosan equals rhamnose multiplied by 0.8. A table giving these calculated figures will be found on page 19 of the article by Ellett and Tollens,^a in which attention is directed to an error in the calculation of 0.028 and 0.029 gram of the methyl-furfural-phloroglucid to rhamnose.

To obtain the weight of pentosans, subtract the final corrected weight of methyl-furfural-phloroglucid from the weight of the mixture of methyl-furfural-phloroglucid and furfural-phloroglucid and calculate according to Kröber's table, or according to the formulas given in the present provisional methods for pentosans.

The following results were obtained by the two chemists engaged in the work:

TABLE I.—*Determination of pentosans and methyl-pentosans.*

GOODRICH'S RESULTS.

Gum tragacanth.	Furfural-phloroglucid + methyl-furfural-phloroglucid.	Loss by alcoholic extraction.	Pentosans.	Methyl-pentosans as rhamnosan.
Grams.	Grams.	Grams.	Per cent.	Per cent.
0.5	0.2258	0.0119	39.42	3.74
0.5	0.2256	0.0125	39.28	3.90
0.5	0.2295	0.0117	40.12	3.69
0.5	0.2185	0.0128	38.00	4.13
Average.....	-----	-----	39.21	3.87
0.8	0.3593	0.0167	38.52	3.11
0.8	0.3544	0.0178	38.11	3.27
0.8	0.3511	0.0170	37.83	3.16
0.8	0.3531	0.0182	37.92	3.35
Average.....	-----	-----	38.10	3.22

^a Jour. Landw., 1905, 53 (1) : 13.

TABLE I.—*Determination of pentosans and methyl-pentosans*—Continued.

HOUGHTON'S RESULTS.

Gum tragacanth.	Furfural-phloroglucid + methyl-furfural-phloroglucid.	Loss by alcoholic extraction.	Pentosans.	Methyl-pentosans as rhamnosan.
Grams.	Grams.	Grams.	Per cent.	Per cent.
0.5	0.2294	0.0194	38.74	5.66
0.5	0.2266	0.0134	39.30	4.12
0.5	0.2320	0.0118	40.54	3.72
0.5	0.2318	0.0142	40.08	4.36
0.5	0.2228	0.0158	38.20	4.74
0.5	0.2346	0.0142	40.08	4.34
0.5	0.2338	0.0138	40.50	4.26
0.5	0.2328	0.0126	40.54	3.92
0.5	0.2358	0.0144	40.76	4.44
0.5	0.2426	0.0166	41.56	4.94
0.5	0.2372	0.0150	40.90	4.54
0.5	0.2356	0.0126	41.04	3.92
0.5	0.2354	0.0116	41.18	3.66
Average.....			40.26	4.36
0.8	0.3592	0.0196	38.43	3.58
0.8	0.3642	0.0216	38.88	3.89
0.8	0.3686	0.0210	39.32	3.79
0.8	0.3672	0.0220	39.10	3.96
0.8	0.3682	0.0182	39.18	3.35
0.8	0.3706	0.0182	39.85	3.35
0.8	0.3517	0.0160	38.01	3.00
0.8	0.3806	0.0236	40.34	4.21
0.8	0.3704	0.0186	39.79	3.41
0.8	0.3746	0.0174	40.35	3.22
0.8	0.3704	0.0158	40.09	2.96
Average.....			39.31	3.52

In interpreting these results, it must be borne in mind that gum tragacanth contains a very large amount of pentosans, larger, in fact, than almost any of the food or feed materials that the chemist will be called upon to examine; hence it was necessary to employ a small original weight of the gum, and this would result in small errors of weighing being very much magnified when the results were expressed on a percentage basis. Judging from Table I the method employed does not give as good results as was hoped. It will at once be seen from the work of both chemists that there is a tendency to get low results on methyl pentosans when the amount of gum tragacanth is increased. Expressing this in another way, there is a tendency for the amount of methyl pentosans to decrease as the weight of the furfural-phloroglucid + methyl-furfural-phloroglucid precipitate increases. This is probably due to the fact that the alcohol extraction is not so complete when a large precipitate is being handled. The results obtained by the same analyst on a definite weight of gum tragacanth do not vary from one another, in the large majority of cases, more than one would expect. The same can not be said of the results obtained by different analysts, which vary from each other more than is desirable even in such a conventional method. However, the averages obtained by the two analysts for methyl pentosans are close enough to warrant the referee for next year in sending out samples for further comparative work.

Even as the method stands it is worthy of adoption by the association as a roughly approximate method to be used by those who desire to make as complete an analysis as possible of vegetable materials. As the analysts become more familiar with the manipulation of the method, which is somewhat difficult and tedious, it is possible that their results will become more uniform. Better results will also be obtained upon the substances usually examined by feeding stuff methods than upon gum tragacanth, in which the amount of furfural-phloroglucid is so large that the extraction of the methyl-furfural-phloroglucid

is somewhat incomplete if a moderate quantity of the original gum is used. I would therefore recommend that the method be further studied next year, especial attention being given to obtaining a large amount of comparative work.

Since calculating the percentages of methyl pentosans (as rhamnosan) from the formula given by Ellett and Tollens, an article by Mayer and Tollens^a has appeared in which is given the formula for calculating the methyl-pentosans to fukose and also a table for calculating methyl-furfural-phloroglucid to fukose, fukosan, rhamnose, rhamnosan, and methyl pentosan (average of fukosan and rhamnosan).

I would, therefore, further recommend that this table be used in calculating the results of analysis, the average figure for methyl pentosans being taken when it is not known whether the mother product is fukosan or rhamnosan.

In accordance with instructions from the association, a study was also made of the time required to dry the crude fat obtained in the ether extract determination on several different feeding stuffs. The dryings were made at the temperature of boiling water and the following feeding stuffs were employed: Wheat germ meal, ground corn, cotton-seed meal, and flaxseed. To be certain that all fat was extracted from the samples, the time of extraction with ether was increased from sixteen to about twenty-four hours. The following results were obtained by the two chemists engaged in the work:

TABLE II.—*Determination of time required to dry the crude fat obtained in ether extracts from different feeding stuffs.*

GOODRICH'S RESULTS.

Material.	Weight of sample.	Crude fat extracted.					
		$\frac{1}{2}$ hour.	1 hour.	$1\frac{1}{2}$ hours.	2 hours.	$2\frac{1}{2}$ hours.	3 hours.
	<i>Grams.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Wheat germ meal.....	1	5.76	4.24	3.89	3.77	3.73	3.73
Do.....	2	4.30	4.00	3.94	3.90	3.88	3.89
Do.....	3	4.03	3.95	3.94	3.92	3.91	3.92
Do.....	4	3.82	3.80	3.86	3.86	3.88	3.89
Do.....	4	3.80	3.76	3.77	3.78	3.80	3.81
Do.....	4	3.77	3.72	3.79	3.80	3.81	3.82
Do.....	4	3.87	3.84	3.89	3.86	3.93	3.92
Do.....	4	3.88	3.85	3.89	3.86	3.93	3.92
Do.....	4	3.86	3.83	3.89	3.86	3.92	3.90
Corn.....	1	4.95	4.76	4.72	4.64	4.69	4.69
Do.....	2	5.69	4.80	4.67	4.61	4.61	4.59
Do.....	3	5.08	4.60	4.49	4.46	4.47	4.47
Do.....	4	4.17	4.16	4.19	4.22	4.23	4.23
Do.....	4	4.22	4.14	4.16	4.17	4.23	4.24
Do.....	4	4.36	4.21	4.22	4.23	4.28	4.31
Cotton-seed meal.....	2	11.44	11.37	11.30	11.33	11.33	
Do.....	2	11.45	11.40	11.29	11.35	11.36	
Do.....	2	11.70	11.50	11.36	11.41	11.43	
Linseed.....	2	27.70	26.85	26.84	26.98	27.02	
Do.....	2	27.38	26.54	26.43	26.45	26.45	

HOUGHTON'S RESULTS.

Wheat germ meal.....	1	4.26	3.92	3.92	4.00	4.02	3.98
Do.....	2	4.45	3.74	3.68	3.65	3.70	3.77
Do.....	3	4.01	3.84	3.83	3.86	3.88	3.92
Do.....	3	4.71	3.91	3.87	3.83	3.85	3.89
Corn.....	1	4.72	4.58	4.80	4.73	4.62	4.62
Do.....	1	6.46	4.36	4.28	4.44	4.42	4.62
Do.....	2	5.19	4.48	4.36	4.44	4.55	4.52
Do.....	3	4.56	4.27	4.23	4.27	4.23	4.25
Do.....	3	4.89	4.33	4.30	4.36	4.36	4.35
Cotton-seed meal.....	1	11.56	11.22	11.22	11.26	11.26	11.34
Do.....	1	12.40	11.24	11.12	11.00	11.06	11.34
Linseed.....	1	27.24	26.62	26.48	26.52	26.54	26.70
Do.....	1	28.60	26.88	27.02	26.92	26.92	26.98
Do.....	1	28.46	26.84	26.64	27.02	27.36	27.24
Do.....	1	27.56	26.38	26.56	26.94	27.18	27.08

^aJour. Landw., July, 55: 269.

It is at once evident from these results that no definite period for drying the crude fat can be specified in the method for determining this substance in cattle food materials. The time necessary to obtain a minimum weight evidently varies with the individual performing the work, with the amount of substance taken, with the character of the fat extracted, with the amount of fat in the substance, and possibly with other factors. A second point brought out is one that would be expected, i. e., that there is a marked tendency for the fat to reach a minimum weight and then slowly to gain by oxidation of the fat. This is, of course, particularly well marked in the case of linseed, which contains an oil that has a high oxygen-absorbing power.

A third point made evident by this investigation is, that in every case at least a one-hour period of drying was necessary to reach a minimum weight, while in some cases this period was extended to two and one-half hours. In the large majority of cases a one and one-half hour period of drying the crude fat was sufficient to obtain either a minimum or results so close to the minimum that the difference was negligible. On the whole, then, while a definite time of drying the fat can not be specified, it may be of some service to the inexperienced to state that the time is usually between one and one-half hours.

I would recommend, therefore, that the last sentence under Crude Fat, or Ether Extract, Direct Method, page 39, Bulletin 107 (Methods of Analysis), which now reads, "Dry the extract at the temperature of boiling water until it ceases to lose weight," be changed to read, "Dry the extract at the temperature of boiling water for one-half hour, remove from the oven to a desiccator, let it cool, and weigh; continue this every alternate one-half hour, drying and weighing until a minimum weight of the fat is obtained. For most substances a period of one to one and one-half hours is required to obtain a minimum weight."

REPORT ON SPECIAL ANALYTICAL METHODS OF SUGAR ANALYSIS.

By C. A. BROWNE, *Referee*.

The work of the referee on special methods of sugar analysis for the present year has covered the investigation of a number of carbohydrate products, but the present report will be limited to a discussion of only three of these: First, the analysis or assay of commercial dextrins; second, a comparison of methods for the estimation of glucose in honey, and third, a study of methods for detecting artificial invert sugar in honey.

ASSAY OF COMMERCIAL DEXTRINS.

During the past year the Bureau of Chemistry has had occasion to analyze a number of dextrins for the work of the Bureau of Engraving and Printing. As a basis for methods an extensive article by Friedrich Lippmann^a upon the "Examination and analysis of commercial dextrins" was used. An abstracted translation of this article has been prepared by the referee, and copies so far as available can be furnished to members of the association.

The methods described in this abstract for moisture, ash, insoluble organic matter, and reducing sugars, were followed, and the difference between these and 100 per cent taken as dextrin, as advocated by Lippmann. It should be noted, however, that this difference includes other cold-water soluble organic materials besides dextrin, such as acids, proteid bodies, and various decomposition products of a carbohydrate nature formed during the process of dextrin-

^a Zts. Spiritus-Ind., 25: 304, 307, 316, 317.

ization. These bodies should not, of course, be included with the dextrin, yet from the fact that there is no satisfactory method for the quantitative isolation of the dextrin from the accompanying impurities, the dextrin is taken as difference. For the specific purpose for which the dextrans are used at the Bureau of Engraving and Printing it was necessary to make a somewhat closer differentiation of the cold-water soluble matter (even if it were only an arbitrary one) which would throw more light upon the character of the products. Therefore the specific rotation of the product was first determined; the specific rotation equivalent to the reducing sugars present as dextrose was then subtracted from this and the remainder taken as the specific rotation due to the pure dextrin present. This remainder multiplied by 100 and divided by 186, the specific rotation of pure dextrin,^a will give the percentage of dextrin present. The difference between the sum of the constituents by this method of calculation and 100 is taken as the undetermined solubles. While the polariscopic determination of dextrin, by such a method as the one described, is of but little scientific value for the reasons given by Lippmann, it is believed that the results obtained by it have a decidedly practical value and give the chemist a more accurate idea of the character of his product than where dextrin is estimated simply by difference.

In the following table results obtained by the method described upon eleven samples of commercial dextrin are given. It will be noted that the results for dextrin obtained from specific rotation are more nearly in accord with the relationship indicated by viscosity than by the estimation of dextrin by difference.

Analysis of dextrans.

Num- ber.	Specific rotation [α] D	Viscosity (1:2 so- lution water = 100).	Moisture 105° C.	Ash.	Reduc- ing sugars as dex- trose.	Cold water, insolu- ble, or- ganic.	Dextrin by dif- ference.	Dextrin from polariz- ation.	Unde- ter- mined soluble.	Acidity N (10 NaOH per 10 grams).
			<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>cc.</i>
1	+157.7	250	6.23	0.07	1.80	0.32	91.58	84.34	7.24	2.0
2	+173.9	425	1.59	.12	2.16	.23	95.90	92.94	2.96	2.4
3	+163.1	275	4.69	.17	4.26	.55	90.33	86.53	3.80	2.4
4	+169.9	338	2.48	.06	3.34	.18	93.94	90.47	3.47	3.5
5	+170.6	450	4.13	.07	2.16	.51	93.13	91.18	1.95	2.5
6	+169.4	450	4.70	.10	1.43	.67	93.10	90.52	2.58	2.5
7	+148.3	200	3.98	.10	6.77	1.25	87.90	77.87	10.03	2.4
8	+171.5	388	2.59	.18	2.15	.44	94.63	91.69	2.94	2.6
9	+157.9	263	3.11	.16	4.59	.72	91.42	83.65	7.77	2.8
10	+163.1	313	5.21	.09	3.05	.44	91.21	86.88	4.33	2.4
11	+162.9	350	7.43	.06	1.85	.98	89.68	87.13	2.55	2.3

The viscosity determination is of paramount value as a physical test, the results by this corresponding most closely with the practical results to which the dextrin is put. Arranging the samples in order of viscosity, it will be seen that the value of a dextrin, as would be expected, is inversely proportional to the amount of undetermined solubles.

The following are offered to the association as provisional methods for the analysis of commercial dextrans:

METHODS OF ANALYSIS.

Physical constants.

Specific rotation.—Transfer 10 grams of the finely divided sample to a 100 cc flask, and after solution in about 50 cc of cold water add 5 cc of alumina cream and make up the contents to 100 cc, thoroughly shake, and filter. Polarize the

^a L. Schulze, J. prakt. Chem., 1883, 28: 327.

filtrate in a 200 mm tube, using an S. and H. type of saccharimeter. Specific rotation $(\alpha)_D = \frac{V \ 34.68}{20}$, in which V =Ventzke reading.

Viscosity.—Dissolve 100 grams of dextrin in 200 cc of cold water and determine the viscosity of the solution by any of the standard forms of viscosimeter. Comparative results should always be made by the same instrument and under similar conditions of temperature. Results are expressed upon the basis that water equals 100.

Chemical analysis.

Moisture.—Determine by drying from 2 to 5 grams of sample for four hours at a temperature of 105° C. Absolute constancy in weight can not be attained on account of the slow decomposition of the dextrin.

Ash.—Determine by the official method of the association, Bulletin 107, Bureau of Chemistry.

Soluble starch.—If a filtered hot-water solution of the dextrin gives a strong blue reaction with iodine, soluble starch is indicated. Weigh two lots of dextrin, 10 grams each, into a 100 cc flask, add 50 cc of cold water to each, and after all soluble matter is dissolved make up the contents of the one flask with cold water to 100 cc, shake, and filter. Evaporate 20 cc of the solution (2 grams) to dryness and dry for four hours at 105°. as under determination of moisture. Weight of residue less ash on incineration equals cold-water soluble organic matter. Heat the contents of the second flask to boiling, and then after cooling make up to 100 cc, shake, and filter. The weight of hot-water soluble organic matter in 20 cc of solution is determined as before. Hot-water soluble organic less cold-water soluble organic gives the soluble starch.

Unconverted starch.—If the residue insoluble in hot water shows under the microscope grains which are colored blue with iodine, unconverted starch is present. To determine the percentage, collect the residue insoluble in hot water on a filter, wash until free from soluble matter, and determine the starch by the usual method.

Reducing sugars.—Determine in an aliquot of the cold-water soluble by the method of Allihn, the results being expressed as dextrose.

Dextrin.—Subtract the specific rotation of the dextrin due to reducing sugars $\left(\frac{52.5 \times \text{per cent reducing sugar as dextrin}}{100} \right)$ from the original specific rotation of the sample. Multiply the remainder by 100 and divide by 186 ($[\alpha]_D$ for pure dextrin) to obtain the calculated percentage of dextrin in the sample.

Undetermined solubles.—The per cent of cold-water soluble organic matter less calculated percentage of dextrin gives the percentage of undetermined solubles.

The other two studies on a comparison of the methods for the estimation of glucose in honey and the detection of invert sugar in honey are reported in full in Bulletin 110 of the Bureau of Chemistry, entitled, Chemical Analysis and Composition of American Honey; they are, therefore, omitted from the Proceedings.

REPORT ON SUGAR AND MOLASSES METHODS.

By C. A. BROWNE, *Referee*, and J. E. HALLIGAN, *Associate Referee*.

The samples sent out for cooperative work by the referees on sugar and molasses consisted of a sample of Louisiana low-grade sugar and one of molasses obtained from Dr. G. L. Spencer, Pijuan, Cuba, representing the final

product of a Cuban sugar house. The following circular letter of instructions was sent to the cooperating chemists:

INSTRUCTIONS FOR ANALYSIS OF SUGAR AND MOLASSES SAMPLES, 1907.

I.—*Moisture.*

The objections raised at the last meeting to the method of drying ten consecutive hours (Bulletin 46, p. 27), thus exceeding the length of the usual laboratory day, renders further work upon the determination of water in molasses necessary. It is recommended that the method of Pellet (International Sugar Journal, 1906, 8: 509) be tested for this purpose. The method reads as follows:

Weigh 3 grams of molasses into a flat-bottom dish containing about 10 grams of finely crushed pumice stone and note the total weight. Dilute the molasses with a little boiling water, add one drop of ammonia and mix. Dry at 102° to 105° C. to constant weight, which will be complete in from two and one-half to three and one-half hours if the drying oven be previously heated to the required temperature.

Compare Pellet's method with the method of drying in vacuo, with any of the official methods, or with any other method preferred by the analyst.

Determine moisture in the sugar by the ordinary methods.

II. *Determination of reducing sugars in molasses and sugar.*

A. Clarify the solutions with lead subacetate in the usual way, removing any excess of lead with sodium carbonate (Bulletin 46, p. 33) or potassium oxalate (Circular 26, page 5).

(1) Determine reducing sugars by the method of Allihn (Bulletin 46, p. 35) and determine the reduced copper:

(a) From weight of Cu_2O .

(b) From weight of CuO (oxidation of Cu_2O after weighing by heating in a muffle).

(c) Volumetrically by Low's method.

The copper from (b) is dissolved in nitric acid and the copper determined either by Low's modified bromin method (Proceedings of 1905, Bulletin 99, p. 19) or the original Low zinc-acetate method. (J. Amer. Chem. Soc., 1902, 24: 1082.)

(2) Determine reducing sugars by method of Munson and Walker. (J. Amer. Chem. Soc., 1906, 28: 663.)

B. Make the same determinations on the filtered solutions without clarification with lead subacetate.

III. *Polarization, direct and invert, of molasses and sugar.*

Weigh out a normal weight and make to 100 cc or to such multiple thereof as may be necessary to secure an accurate polarization after clarifying as follows:

(a) With lead subacetate solution (Bulletin 46, pp. 38–39).

(b) With Horne's dry lead subacetate (J. Amer. Chem. Soc., 1904, 26: 186).

(c) With alumina cream and a little sodium hydrosulphite (B. A. S. F. preparation the best).

If there is time, compare the sucrose determination by double polarization with the gravimetric method.

Record carefully all temperatures of polarization, dilutions, etc., that results may be compared on as uniform a basis as possible.

It is also urged that the work on the samples be begun if possible immediately upon their arrival to avoid any changes in composition which might result from fermentation.

C. A. BROWNE, Jr., *Referee on Sugar.*

J. E. HALLIGAN, *Associate Referee on Molasses Methods.*

SUGAR.

The cooperative work on sugar for the year consisted in a comparison of different methods of clarification; first, as to their effect on the direct and invert polarization; and second, as to their effect on the determination of reducing sugars.

The results of the different chemists on the polarizations of sugars are given in the following table:

TABLE I.—*Polarization of sugar with different methods of clarification.*
[Normal weight to 100 cc, 200 mm tube.]

Clarifying agent and analyst.	Amount of clarifying agent.	Direct polarization.	Corrected invert polarization.	Temperature of polarization.	Sucrose by Clerget's method (factor 142.66).
Subacetate of lead:	cc.	° V.	° V.	° C.	Per cent.
A. W. Blair, Florida.....	5	82.6	-26.8	31	86.03
C. A. Browne, Washington, D. C.....	5	82.8	-29.6	26	86.68
A. H. Bryan, Washington, D. C.....	6	82.8	-32.0	20	86.53
A. Given, Washington, D. C.....	6	83.0	-30.0	20	85.18
J. E. Halligan, Louisiana.....	7	82.5	-26.6	29	85.13
W. D. Horne, New York ^a	6	82.0	-28.8	24.5	84.96
W. P. Naquin, Louisiana.....	5	83.1	-28.1	31	87.45
M. H. Wiley, Washington, D. C.....		82.7	-27.7	29	86.14
Fritz Zerban, Louisiana.....	5	83.2	-30.6	25	87.43
Average.....		82.84			86.32
Dry subacetate of lead:	Grams.				
C. A. Browne, Washington, D. C.....	1.0	82.4	-29.3	26	86.12
A. H. Bryan, Washington, D. C.....	2.0	82.8	-32.7	20	87.02
A. Given, Washington, D. C.....	2.0	82.7	-28.0	20	83.45
J. E. Halligan, Louisiana.....	1.5	82.5	-26.4	30	85.30
W. D. Horne, New York ^a	0.8	81.6	-27.6	24	83.62
W. P. Naquin, Louisiana.....		82.6	(b)		
M. H. Wiley, Washington, D. C.....	1.0	82.6	-28.2	28.5	86.52
Fritz Zerban, Louisiana.....		83.0	-30.8	25	87.43
Average.....		82.66			85.97
Neutral lead acetate:	cc.				
C. A. Browne, Washington, D. C.....	2.5	82.1	-29.2	26	85.80
A. H. Bryan, Washington, D. C.....	5	82.4	-32.2	20	86.39
J. E. Halligan, Louisiana.....		82.0	-25.6	30	84.29
M. H. Wiley, Washington, D. C.....	5	82.5	-28.8	29	86.81
Average.....		82.25			85.82
Alumina cream and sodium hydrosulphite:	Grams.				
C. A. Browne, Washington, D. C.....	0.1	81.9	-29.4	26	85.82
A. H. Bryan, Washington, D. C.....	0.5	81.4	-32.9	20	86.16
A. Given, Washington, D. C.....	0.3	82.5	-30.2	20	84.96
J. E. Halligan, Louisiana.....	0.5	81.7	-26.8	30	85.00
W. P. Naquin, Louisiana.....		82.8	-27.0	32	86.69
M. H. Wiley, Washington, D. C.....		82.4	(b)		
Fritz Zerban, Louisiana.....		82.8	(c)		
Average.....		82.21			85.73

^a Results of W. D. Horne omitted from average; the sugar was not analyzed immediately on its arrival and had evidently deteriorated through fermentation.

^b Too dark to read.

^c Precipitation of sulphur prevented readings.

As regards direct polarization, the results of the different chemists, considering the variations of temperature, etc., under which the work was performed, show a fairly uniform agreement with each method of clarification. The percentages of sucrose by the Clerget method, however, show extremely wide variations. These are due, no doubt, partly to differences in the method of inversion and also to variation in dilution of the inverted solution, which was always darker. There is probably no determination where the element of personal error or equation is more evident than in the Clerget method for sucrose. This fact and the numerous inherent errors in the method has led Koydl ^a, in a recent article, to recommend the complete abandonment of the Clerget process in analytical work and the adoption of the gravimetric process.

Comparing the direct polarizations, which have the greatest interest from a commercial point of view, a number of interesting facts are noted in connection

^a Chem. Ztg., 1907, 31: 339.

with the different methods of clarification. But before taking these up it may be well to refer briefly to several of the factors which have a bearing on the subject. The true polarization of a raw sugar is represented by the sum of the polarizations of the various optical constituents present, which include not only sucrose, but also the reducing sugars, dextrose, levulose, and other products, such as caramel, amids, amino acids, etc. The differences obtained by the use of different clarifying agents are due partly to the selective action of the precipitant upon the optical bodies other than sucrose and also partly to the volume of the precipitate. In the table it is seen that the average of the results when the subacetate of lead solution is used is 0.15 per cent higher than that obtained with the dry subacetate. This difference, while not noticeable in the work of each analyst, is nevertheless well marked and is to be explained by the greater concentration of the solution by the volume of the lead precipitate. Comparing these solutions with the one clarified with neutral lead acetate, we note that the latter gives a polarization decidedly lower, notwithstanding the volume of precipitate error. This is to be explained by the fact that subacetate of lead, whether wet or dry, is a powerful precipitant of the reducing sugar levulose, thus increasing the dextrorotation. Neutral acetate of lead has little if any action upon the reducing sugars.

The lowest readings of all were obtained with alumina cream and hydrosulphite of soda, and these readings ought to represent most closely the true polarization of the sugar, since there is no volume of precipitate error and no precipitation of reducing sugars. Hydrosulphite, however, if allowed to act for any length of time, seems to lower the polarization of some of the component sugars, though just how this is brought about is uncertain. With sucrose it seems to be due to a slight inversion, with dextrose it may result from the formation of sulpho-compounds of lower specific rotation. The referee has deferred the addition of the hydrosulphite until just before polarizing, adding a few small crystals to the solution in the tube before making the reading. An instantaneous decolorization is produced, and a reading can be taken before the sugars are appreciably acted upon. This can be done even in the inverted solution, the liquid remaining clear for five minutes before turbidity begins through decomposition of the hydrosulphite.

The effect of different clarifying agents upon mixed solutions of sucrose, dextrose, and levulose, in the presence of inorganic and organic matter precipitable by lead has been studied at the Bureau of Chemistry by Mr. A. H. Bryan. The work is not yet completed but the results of one set of experiments are given in the following table:

TABLE II.—*Polarization of mixtures of sucrose, dextrose, and levulose with 0.5 gram ammonium oxalate and 0.5 gram sodium sulphate using different clarifying agents.*

No.	Amount of clarifying agent.	Clarifying agent.	Direct polarization.
	cc.		Per cent.
1	5	Alumina cream.....	89.00
2	3.5	Lead subacetate solution.....	89.50
3	7	do.....	89.55
4	3	Normal lead acetate solution.....	89.20
5	6	do.....	89.20
6	a 1.5	Dry lead subacetate.....	89.05
7	4	Alkaline lead nitrate solution.....	89.00
8	a 1	Sodium hydrosulphite.....	88.60

a Grams.

Taking the experiment with alumina cream as the true polarization, it is seen that the lead subacetate solution gives a reading 0.5 per cent too high, and the normal acetate 0.2 per cent too high. The excess reading in the second experiment is due to volume of precipitate and in the first to both volume of precipitate and precipitation of levulose. The dry lead and alkaline lead nitrate give readings in this particular experiment exactly identical with the true polarization. This might seem at first sight to indicate no precipitation of optically active bodies; such a precipitation does take place, however, as will be shown later, and the experiment only shows that in this particular instance the dextrose and levulose were precipitated together in quantities sufficient to neutralize one another.

The determination of reducing sugars in the sugar by the method of Allihn and of Munson and Walker are given in the following table:

TABLE III.—*Determination of reducing sugar in sugar as dextrose by Allihn's and by Munson and Walker's method, using different clarifying agents.*

Clarifying agent and analyst.	Strength of solution.	Precipitant.	Weighing as Cu_2O .		Weighing as CuO .		Titration of copper. (Low's method.)	
			Allihn method.	M. and W. method.	Allihn method.	M. and W. method.	Allihn method.	M. and W. method.
None:			<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
A. H. Bryan.....	26 gm. to 100.		6.45	6.29	6.22	5.98	5.88	5.83
J. E. Halligan.....	1 gm. to 100.		6.94					
W. D. Horne ^a	4 gm. to 100.		7.08	6.43	7.05	6.51	7.02	6.37
Average.....			6.82	6.36	6.63	6.25	6.45	6.10
Subacetate lead:								
A. W. Blair.....	{ 10 gm. to 200. 100 cc to 200.	{ 2½ cc..	6.33	5.77				
A. H. Bryan.....	{ 26 gm. to 100. 10 cc to 100.	{ 6 cc..	6.14	5.76	5.67	5.51	5.67	5.30
J. E. Halligan.....	{ 1 gm. to 100. 1 gm. to 100.	{ 2 cc..	6.65					
W. D. Horne ^a	{ 1 gm. to 100. 26 gm. to 100. 25 cc to 100.	{ 4 cc.. 6 cc..	6.44 6.61	 6.19	 6.51	 6.01	 6.51	 5.99
Average.....			6.43	5.91				
Dry subacetate lead:								
A. H. Bryan.....	{ 26 gm. to 100. 10 cc to 100.	{ 3 gr..	6.05	5.75	6.00	5.72	6.02	5.62
J. E. Halligan.....	{ 1 gm. to 100. 1 gm. to 100.	{ ½ gr..	6.67					
Average.....			6.36					
Neutral lead:								
J. E. Halligan.....	1 gm. to 100.	2 cc..	6.92					

^a The high determinations of Mr. Horne are probably due to a slight deterioration of the sugar through fermentation.

The highest results are obtained when no clarifying agent is used or only neutral acetate of lead; the results after clarifying with subacetate of lead solution or dry subacetate of lead are about 0.5 lower. The French chemists, and more lately the English chemists, have declared against the use of basic lead acetate in clarification for the determination of reducing sugars, and their contention seems wholly justified. To determine the extent of this error in analytical work the following series of experiments were carried out at the Bureau of Chemistry by Mr. A. H. Bryan. Solutions of dextrose and levulose were prepared, using 5 grams of sugar and 1 gram each of magnesium sulphate and ammonium tartrate. To 50 cc of this solution the precipitant was added and the volume made up to 100 cc. After filtering the excess of lead was removed with potassium oxalate and the sugar determined by Allihn's method.

TABLE IV.—Percentages of dextrose and levulose removed by different precipitating agents (A. H. Bryan).

[1 gram of magnesium sulphate and 1 gram of ammonium tartrate added to each 5 grams of sugar in solution.]

Precipitating agent.	Amount per 100 cc of solution.	Dextrose removed.	Levulose removed.
	cc.	Per cent.	Per cent.
Normal lead acetate solution.....	3.5	0.93	0.00
Do.....	7.0	0.84	0.00
Lead subacetate solution.....	3.5	3.35	8.03
Do.....	7.0	8.34	19.91
Dry lead subacetate.....	^a 1.0	3.85	14.93
Do.....	^a 2.5	17.48	35.33
Alkaline lead nitrate (Herle's solution).....	4.0	6.27	13.84
Do.....	8.0	5.61	25.12

^a Grams.

The normal acetate of lead removes practically no reducing sugar in any of the experiments; the lead subacetate, on the other hand, in any of its forms, removes very large quantities of both dextrose and levulose, the latter sugar always in much larger amounts. In our present official methods for reducing sugars in saccharine products, wines, fruits, etc., the subacetate of lead is always prescribed for clarification. In view of the manifest error involved in the use of this reagent, it is recommended that its use be discontinued by the association in the determination of reducing sugars.

The results obtained by the different methods of estimating copper show, as was indicated last year, the highest result by the method of weighing as cuprous oxid (Cu_2O), and the lowest result by the Low method of titration. This is due to the contamination of the cuprous oxid with slight amounts of organic and mineral matter. With products of very low purity it is always safer to make a direct determination of the copper than to weigh the precipitated oxid.

MOLASSES.

DETERMINATION OF TOTAL SOLIDS.

The comparison of methods upon total solids made last year was again continued, the object of the cooperative work being to find a simple and rapid method which would give results agreeing closely with the method of drying in vacuo.

The results of the work are given in the following table:

TABLE V.—Determination of total solids in molasses.

Analyst	1 gram at 98°.			2 grams, at 98°.	2 grams, at 105°.	Pellet method, 103°.		Vacuum.	
	2 hours.	3 hours.	4 hours.	10 hours.	6 hours.	3½ hours.	5 hours.	98°, 7½ hours.	To constant weight.
A. H. Bryan, Washington.....	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.	P. ct.
J. E. Halligan, Louisiana.....				75.39		77.60	74.21		^a 76.87
W. D. Horne, New York.....			75.47	75.55	74.93	74.64		75.54	
M. M. Klaverweiden, Cuba.....				74.89					
W. P. Naquin, Louisiana.....				76.76					
G. L. Spencer, Cuba.....	76.29	76.24							
A. Wedderburn, Cuba.....									76.70
F. Zerban, Louisiana.....				76.36					
Average.....	76.29	76.24	75.47	75.79	74.93	76.12	74.21	75.54	76.79

^a 38 hours at 70° C.

In commenting upon the above, W. D. Horne remarks that the Pellet method of heating to 105° gives too high results owing to caramelization.

J. E. Halligan, in his method of drying six hours at 104° in a salt-water bath, obtained results agreeing closely with the method of drying 2 grams ten hours at 98°.

Mr. Bryan and Mr. Wedderburn by the method of drying in vacuum to constant weight obtained closely agreeing results, and the figures by this method, according to Mr. Spencer, are in best concordance with the workings of the sugarhouse.

The polarizations of the molasses by the different chemists using various methods of clarification are given in the following table:

TABLE VI.—*Polarisation of molasses with different methods of clarification.*

Clarifying agent and analyst.	Half normal weight to—	Amount of clarify- ing agent.	Polarization.				Sucrose by Clerget method (factor 142.66).
			Direct.		Invert.		
Subacetate of lead filtered and acetic acid added until neutral or slightly acid:	cc.	cc.	°P.	°C.	°P.	°C.	°P.
J. E. Halligan, Louisiana.....	100	11.0	25.6	30.0	—20.0	30.0	35.72
W. D. Horne, New York.....	200	a50.0	25.2				
M. Klaverweiden, Cuba.....	100	20.0	26.6				
W. P. Naquin, Louisiana.....	200	9.5	26.0	30.0	—17.6	31.0	34.29
G. L. Spencer, Cuba.....	100	20.0	26.6		—19.0	23.5	34.85
Fritz Zerbán, Louisiana.....	200	9.5	27.2	28.5	—16.8	28.0	34.19
Average.....			26.2				34.76
Subacetate of lead polarized without acetic acid:							
A. H. Bryan, Washington.....	200	8.0	24.4	20.0	—24.6	20.0	36.95
W. D. Horne, New York.....	200	a50.0	26.0	26.0	—18.4	26.0	34.24
M. Klaverweiden, Cuba.....	100	20.0	28.6				
G. L. Spencer, Cuba.....	100	20.0	28.8		b—19.2	23.7	36.69
Average.....			26.95				35.96
Dry subacetate of lead:		Grams.					
A. H. Bryan, Washington.....	100	3.0	25.0	20.0	—20.7	20.0	34.45
J. E. Halligan, Louisiana.....	100	6.0	28.3	30.0	—17.5	30.0	35.87
W. D. Horne, New York.....	200	6.74	26.2	22.0	—19.2	22.0	34.48
W. P. Naquin, Louisiana.....	200		25.6	30.0	—18.4	30.0	34.46
Fritz Zerbán, Louisiana.....	200		(c)				
Average.....			26.27				34.81
Hydrosulphite and alumina cream:							
A. H. Bryan, Washington.....	200	3.0	18.8	20.0	—26.4	20.0	34.07
J. E. Halligan, Louisiana.....	200	.5	23.8	30.0	—18.9	30.0	33.44
W. D. Horne, New York.....			(c)				
W. P. Naquin, Louisiana.....			(c)				
Fritz Zerbán, Louisiana.....	200		27.2	29.0			
Average.....			23.27				33.76
Hydrosulphite added after subacetate of lead:							
A. H. Bryan, Washington.....	100		20.6	20.0	—24.2	20.0	33.77
M. Klaverweiden, Cuba.....	100		25.6		(c)		
G. L. Spencer, Cuba.....	100		27.0		(c)		
Average.....			24.4				33.77

a 24 Brix lead subacetate.

b Lead not removed prior to inversion (Prinsen-Geerligs method).

c Too dark to read.

In the factory method employed by G. A. Spencer in Cuba, one-half the normal weight is made up to 100 cc after clarifying with lead subacetate solution (54.3° Brix), using as little as will give a suitable solution; 50 cc of the filtered

solution are acidulated with dilute acetic acid to decompose the soluble levulose of lead, and the volume is completed to 55 cc. The polariscopic reading, increased by one-tenth, is multiplied by 2 to give the corrected direct reading. Doctor Spencer states that the direct polarization is always too high when the excess of lead is not removed or neutralized with acetic acid.

In the use of potassium oxalate for removing lead, a number of the analysts experienced the difficulty of turbid filtration. W. D. Horne states that this difficulty can be obviated by the use of a little alumina cream.

The results confirm the observations made on the polarization of the raw sugars, namely, that higher results were secured with the wet subacetate of lead than with the dry and that the lowest results were obtained with the hydrosulphite. The question of the influence of the several clarifying and decolorizing agents has already been discussed under sugar and need not be referred to again here. The variations in the direct polarization of the molasses in each series of experiments show in many instances exceedingly wide variations, a circumstance due largely to difference in the temperature of polarization. These differences, it will be noted, are largely equalized in the calculations of the sugar by the Clerget formula, where the variations between the different chemists are less pronounced. The reverse of this was the case in the analysis of the sugar. The exceedingly large dilution necessary to secure a clear reading (in some cases the results had to be multiplied by 16), of course, magnifies greatly any slight error in the initial observations.

Regarding the use of hydrosulphite as a bleaching agent, many of the chemists reported a difficulty in securing a sufficiently clear reading with the use of this substance alone. There appeared to be an insufficient decolorization in some instances, while in other cases there was a redarkening of the bleached solution due to oxidation. Weisberg ^a has recently studied the action of hydrosulphites, and concludes that the action is a double one, first, by means of the free sulphurous acid where the bleaching action is permanent, and secondly, by means of the nascent hydrogen which is evolved, when there is a redarkening of the solution through oxidation. The referee has found that this after-darkening may be prevented by the use of a new hydrosulphite derivative, sodium sulfoxylate-formaldehyde, sold commercially as Rongalite C. Hydrosulphite bleaches are being used extensively in sugar work just at present, and the effect of these upon the polarization and reducing power of various sugars are points which should be thoroughly investigated by the referee next year.

The gravimetric determination of the sucrose in the molasses was made by Mr. A. H. Bryan with the following results:

TABLE VII.—*Gravimetric determination of sucrose in molasses by Allihn's, and by Munson and Walker's, method, using different clarifying agents.*

Clarifying agent.	Allihn method.			Munson and Walker method.		
	Weighing as Cu ₂ O.	Weighing as CuO.	Titration of Cu by Low's method.	Weighing as Cu ₂ O.	Weighing as CuO.	Titration of Cu by Low's method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
None.....	33.80	33.98	34.68	33.56	34.19	33.66
Lead subacetate.....	34.63	34.75	35.80	33.56	33.84	34.79
Dry subacetate of lead.....	36.46	34.87	33.68	35.07	33.42	32.41

Centrl, Zuckerind., 1907, 15: 975.

The determinations of reducing sugars in the sample of molasses are reported in the following table:

TABLE VIII.—*Determination of reducing sugar as dextrose in molasses by Allihn's, also Munson and Walker's, method, using different clarifying agents.*

Clarifying agent and analyst.	Quantity of precipitant used.	Allihn method.			Munson and Walker method.		
		Weighing as Cu ₂ O.	Weighing as CuO.	Titration of Cu by Low's method.	Weighing as Cu ₂ O.	Weighing as CuO.	Titration of Cu by Low's method.
None:	cc.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
A. H. Bryan, Washington.....		19.77	19.37	19.45	19.20	18.34	18.43
J. E. Halligan, Louisiana.....		20.68					
W. D. Horne, New York.....		20.60	20.06	19.97	20.00	19.43	19.44
Fritz Zerban, Louisiana.....		19.39	19.22	19.62	19.12		
Average.....		20.11	19.55	19.68	19.44	18.89	18.94
Subacetate of lead:							
A. H. Bryan, Washington.....	5	17.51	16.47	16.29	17.27	16.26	15.97
J. E. Halligan, Louisiana.....	5	19.55					
W. D. Horne, New York.....	5	19.45	19.16	19.16	19.00	18.53	18.26
Fritz Zerban, Louisiana.....	5	17.45		17.76	17.87		
Average.....		18.49	17.82	17.74	18.05	17.39	17.12
Dry subacetate of lead:							
A. H. Bryan, Washington.....	$\alpha \frac{1}{2}$	17.85	17.55	17.57	17.60	17.32	17.27
J. E. Halligan, Louisiana.....	$\alpha \frac{1}{2}$	19.09					
Average.....		18.47	17.55	17.57	17.60	17.32	17.27
Neutral lead:							
J. E. Halligan, Louisiana.....	4	19.99					

α Gram.

VOLUMETRIC METHOD.

Analyst.	Soxhlet method.		Violette method.	
	Clarification.	Dextrose.	Clarification.	Dextrose.
J. E. Halligan.....	None.....	Per cent. 20.40		Per cent.
G. L. Spencer.....			Normal lead..	17.55

The results confirm the observations in the case of sugar, showing that a large amount of reducing sugars is precipitated by basic lead. The slightly higher result obtained in each instance by the Allihn method as compared with that of Munson and Walker is explained by the greater inverting action of the Allihn solution upon the sucrose.

THE EFFECT OF HYDROSULPHITE AND RONGALITE ON THE POLARIZATION OF DEXTROSE, LEVULOSE, AND SUCROSE.

By A. H. BRYAN.

Solutions of 10 grams of the pure sugars in 100 cc of water were prepared and to aliquot parts (25 cc) were added one-fourth, one-half, and 1 gram of the bleaching agents. When these salts were dissolved by shaking, the solutions were made up to 50 cc with water and polarized immediately. The tubes containing the solutions were set aside and polarized after five hours and again

after standing one day. Care was taken to have all the solutions of the same temperature. The results of polarization are contained in the following table:

Determinations of the effect of sodium hydrosulphite B. A. S. F. and rongalite C. on the polarization of dextrose, levulose, and sucrose.

Bleaching agents.	Dextrose.			Levulose.			Sucrose.		
	Imme- diate polar- ization.	After 5 hours.	After 24 hours.	Imme- diate polar- ization.	After 5 hours.	After 24 hours.	Imme- diate polar- ization.	After 5 hours.	After 24 hours.
Original polariza- tion.....	+15.4	+15.3	+15.3	-25.0	-25.0	-25.25	+19.2	+19.2	+19.1
With a crystal or two in polariza- tion tube.....	+15.25	+15.1	+15.0	-25.05	-----	-----	+19.2	-----	-----
One-fourth gram hydrosulphite.....	+15.1	Cloudy.	+14.85	-25.0	-25.1	-25.35	+19.2	Cloudy.	+18.95
One-half gram hy- drosulphite.....	+15.1	Cloudy.	+14.55	-25.1	Cloudy.	-25.5	+19.1	Cloudy.	+18.9
1 gram hydrosulph- ite.....	+15.1	+14.15	+13.75	-25.1	-25.2	-25.5	+19.1	+19.05	+19.05
One-fourth gram rongalite.....	+15.35	+15.3	+15.1	-25.05	-25.2	-25.25	+19.2	+19.05	+19.05
One-half gram ron- galite.....	+15.4	+15.3	+15.1	-25.05	-25.2	-25.3	+19.2	+19.0	+19.05
One gram rongalite.	+15.4	+15.35	+14.95	-25.1	-25.2	-25.4	+19.1	+19.1	+19.1

Hydrosulphite has more effect on the polarization of the sugars than rongalite, and lowers the polarization of dextrose more than that of the other two sugars. With sucrose there is a change in the immediate polarization on the addition of 0.5 gram, but on standing one day, 0.25 gram reduces the rotation. Whether this action is due to an inversion of the sucrose, it is impossible to say at present. With levulose there seems to be a slight increase in the polarization, but in other experiments with this sugar no change in the rotation was noted. Rongalite has a slight action on the rotation of dextrose, but on sucrose and levulose there is apparently no action.

In regard to bleaching, hydrosulphite is much faster than rongalite, but it has the disadvantage that its decolorizations are not altogether permanent, and on standing a very short time sulphur is precipitated, making the liquid too cloudy for a reading. More work along this line is being done at the Bureau of Chemistry.

THE DETERMINATION OF SULPHUROUS ACID IN MOLASSES.

By FRITZ ZERBAN and W. P. NAQUIN.

At the last meeting of the association, in 1906, W. D. Horne called attention to several sources of errors in the provisional official method for the determination of sulphurous acid in foodstuffs when applied to sugar products. In the meantime, the food law has been enacted, and therefore an investigation into this question could no longer be deferred. The different methods that have heretofore been used were studied and compared. The work has not yet been completed, but it was deemed advisable to present the data collected so far mainly for the information of those who have to make determinations of sulphurous acid in sugar products. The work will be published later in full.

The following questions had to be studied: (1) Whether molasses contains any hydrogen sulphid or gives rise to it when boiled in the presence of free acid; and whether hydrogen sulphid and sulphurous acid can be given off

simultaneously without acting upon each other. (2) Whether there is any error due to outside sources (use of gas for heating, rubber for connections, tin for condensers). (3) Whether any volatile substances are given off during the distillation which are apt to be acted upon by iodine; and whether or not iodine is lost by volatilization. (4) Whether the use of a current of carbon dioxide is necessary. (5) Whether the total amount of sulphurous acid distills over with the first half of the distillate.

According to our present knowledge of the composition of the sugar cane and its products most, if not all, of the organic sulphur is in the form of proteid sulphur. By boiling in an acid solution the proteids are decomposed, and so far the following cleavage products containing sulphur have been isolated: Cystine, cysteine, α - and β -thio-lactic acid, thio-glycolic acid, ethylsulphide, methylmercaptan, and hydrogen sulphide. The thio acids mentioned decompose further upon prolonged heating, yielding hydrogen sulphide. The presence of hydrogen sulphide in the distillation products of molasses is therefore to be expected. This was suggested by W. D. Horne and also by Jerome Alexander in a paper on the determination of sulphurous acid in gelatin. The following experiments were performed to decide this question: Fresh cane juice was boiled down to sirup without the use of any chemicals; 100 grams were diluted to 400 cc, 5 cc of glacial phosphoric acid added, and distilled. The distillate was collected in a flask containing de Koninck's reagent for hydrogen sulphide (mercuric cyanide and ammonium chloride in water). A black precipitate was obtained which after filtration was dissolved in bromine water. The solution gave a precipitate of barium sulphate when tested with barium chloride. Experiments carried out in a similar manner with silver nitrate and lead acetate solutions gave the same results. After receiving a copy of Doctor Horne's paper, cadmium chloride was used and our observations could be confirmed. The filtrate from the cadmium sulphide was also tested for sulphur, with positive results. The volatile sulphur which is not in the form of hydrogen sulphide may be due to the volatilization of substances of a mercaptan-like character. We have not been able so far, on account of the small quantities obtained, to identify them or to prevent their determination together with sulphurous acid.

Similar experiments were performed with a molasses obtained by the sulphitation process as practiced in Louisiana. Hydrogen sulphide was again obtained, which proves that under the conditions of procedure sulphurous acid and hydrogen sulphide may coexist. It is, therefore, advisable to use a solution of cadmium-chloride in the determinations, as recommended by Horne.

Gas can be used for heating the distilling flask without any risk. But if it be found necessary to concentrate the distillate, this should be done in flasks with narrow openings. Rubber connections should be avoided as much as possible, especially at that side of the condenser which is connected with the receiver containing iodine or bromine. It was found that the use of rubber may introduce considerable error. When two glass tubes are to be connected with rubber hose the two ends of the glass tubes should touch. In a number of distillations a Kjeldahl condenser with tin serpentines was used, and in this case a slight deposit of sulphur was invariably noticed in the glass tube extending into the receiver. When glass condensers were substituted for those of tin that deposit could not be observed. The deposit is probably due to the interaction of hydrogen sulphide and sulphur dioxide, but the tin seems to play some part in this reaction.

Doctor Horne called attention to the possibility that sugar products like molasses when distilled in acid solution might give off volatile organic substances which are acted upon by iodine. In order to test this question, a sample

of molasses was secured from a house where no sulphur is used. In one experiment the distillate was collected in bromin water after washing with cadmium-chlorid solution, and the sulphur determined gravimetrically in the cadmium sulphid as well as in the filtrate from it combined with the bromin water. In a second determination the official method was used, the dilution in the distilling flask and the quantity of liquid distilled over being the same as in the first experiment. It was found that the official method yielded considerably more "sulphur" than was obtained by the gravimetric determination. However, this discrepancy is partly due to the fact that in spite of the U tube provided for in the official method some iodine volatilizes during the distillation. This could easily be shown by connecting the U tube with a glass tube extending into a small flask which contained a solution of potassium iodide. When the distillation was finished the potassium iodide had taken up a considerable quantity of iodine, not enough, however, to make up for the difference from the gravimetric method.

Several experiments conducted with and without the use of a current of carbon dioxide proved the necessity of using this precautionary measure, although the influence was not so great as we had anticipated. The inlet tube for the carbonic acid gas must extend into the liquid to prevent it from distilling back into this same tube.

After these preliminary experiments the following mode of procedure was adopted, avoiding the errors recognized so far: Transfer 50 grams of molasses to a flask of sufficient capacity and make up to 400 cc with air-free water. Close the flask with a rubber stopper (rubber can safely be used at this point) having three perforations. One of these is for the inlet tube for the carbon dioxide, the second for a small separatory funnel filled with glacial phosphoric acid, the third for a catch-all connected by means of a good cork with a Liebig condenser. Connect the other end of the condenser with a glass tube leading into a filtering flask containing a small quantity of a 2 per cent solution of cadmium chloride. The glass tube must terminate below the surface of the liquid. After the filtering flask follows a U tube containing a little water and then a glass tube leading into bromin water in a little Erlenmeyer flask. The U tube prevents sucking back of the bromin water into the cadmium solution. After the receivers have been put in place, carbon dioxide is passed through the entire apparatus for about ten minutes. Then about 5 cc of phosphoric acid are added from the separatory funnel and 200 cc of the liquid distilled over. When the distillation is finished, the bromin water in the Erlenmeyer and the contents of the U tube are transferred to a beaker, and the cadmium solution containing the distillate is filtered into the bromin water. The excess of bromine is boiled off and the sulphuric acid is precipitated as barium sulphate and weighed. If the hydrogen sulphide is to be determined also, the cadmium sulphide is dissolved in bromin water and the solution treated like the others.

It was deemed inadvisable to distil to a smaller volume than 200 cc in order to avoid charring of the mass at certain points and subsequent reduction of sulphuric acid.

Finally the influence of concentration was studied and very surprising results obtained. When the same quantity of molasses was diluted in one case to 600 cc and in the other to 400 cc, both being distilled down to 200 cc, the first experiment gave a much higher result than the second. This experience led us to investigate how much of the liquid must be distilled to obtain the entire quantity of SO_2 . It was found that by adding again 200 cc of water after having distilled from 400 to 200 cc new quantities of sulphurous acid were obtained, and it was necessary to repeat this procedure four times to distil

over practically all of the SO_2 . In this case the last distillate contained only traces of sulphur. It was thus shown that by distilling over 800 cc the SO_2 could be satisfactorily determined. The method would be facilitated by making up 50 grams of molasses to 1,000 cc and distilling down to 200 cc. In this case the receivers had naturally to be changed to some extent. Results agreed practically with those obtained by separate distillations. After the true amount of SO_2 in a certain molasses had been determined in this way, the official method was tested for its accuracy. By distilling in the same way from 1,000 cc to 200 cc of a N/10 iodine solution for the collection of the distillate and titrating with thiosulphate, results considerably higher than those by the above method were obtained.

The necessity of distilling off 800 cc of liquid renders the method very tedious, and it would be desirable to devise one which requires less time and labor. It is possible that the sulphurous acid may be determined in some other way than distillation. Direct titration is impossible on account of the dark color of the products under examination, but some method of precipitation may directly or indirectly accomplish the purpose. It would also be desirable to find some method by which the different forms of sulphur dioxide in molasses could be distinguished.

Mr. Wiley called attention to the value and timeliness of Mr. Zerban's paper, inasmuch as both State and Federal food officials are concerned with the difficulties in distinguishing between natural sulphur and added sulphites.

Upon motion by Mr. Wiley, election of officers was made special order for 10 o'clock Friday; and on motion by Mr. Van Slyke reports of all special committees, except the committees on recommendations of referees, were made special order for 11 o'clock.

The association adjourned for the day.

THIRD DAY.

FRIDAY—MORNING SESSION.

REPORT ON MEDICINAL PLANTS AND DRUGS.

By L. F. KEBLER, *Referee*.

The burden of other work imposed upon the staff of the drug laboratory and other pharmaceutical chemists of the country in consequence of recent food and drug legislation, both National and State, has interfered somewhat with the prosecution of the cooperative work on drug assaying during the past year. It is hoped that when the most urgent problems have been solved the study of methods will receive an amount of attention commensurate with its fundamental importance. In the meantime it has been deemed advisable not to depend upon the cooperation of chemists generally in the work, but to continue the same within the drug laboratory as circumstances permit until the time is more propitious for general participation.

In view of the legal status conferred upon the United States Pharmacopœia, Eighth Revision, it seemed desirable to study all of the plant drug assays official therein, comparing them with some of the best available nonofficial methods. After this work was begun, the committee of revision of the Pharmacopœia promulgated two lists of additions and corrections under date of May 1 and June 1, 1907, which introduced some minor changes in the details of certain methods. These changes were recognized in the work subsequently done.

The work this year covered methods for the assay of aconite leaves, aconite root, belladonna leaves, belladonna root, cinchona bark (yellow and red), coca leaves, colchicum corm and colchicum seeds. Samples of these drugs, delivered as being of United States Pharmacopœia quality, were made the basis of study, and the following programme of work was pursued by three collaborators in the drug laboratory. All calculations and solutions, except as otherwise specified, were based on the data of the United States Pharmacopœia, Eighth Revision.

DIRECTIONS FOR THE WORK.

VOLUMETRIC SOLUTIONS.

Prepare a standard normal sulphuric acid, determining the factor by two or more of the following methods:

(1) The United States Pharmacopœia method by titration with normal potassium hydroxid solution which has been standardized against purified potassium bitartrate.

(2) By titration of the ignition residue from a weighed portion of potassium bitartrate.

(3) By titration of a weighed amount of freshly ignited sodium carbonate.

(4) By precipitation as barium sulphate and gravimetric determination.

(5) By evaporation with slight excess of ammonium hydroxid, and determination after drying at 125° C. as ammonium sulphate.

From this normal acid prepare a decinormal sulphuric acid by dilution.

Prepare a fiftieth normal solution of potassium hydroxid and determine the factor by titration with the decinormal sulphuric acid prepared as just directed.

DETERMINATION OF ALKALOID.

Total extraction method.

Into a 200 cc flask weigh 10 grams of the powdered drug, add about 75 cc of ether-chloroform mixture (5 to 1 by volume), rotate and add 5 cc of a mixture

of 40 cc of strong ammonia water with 60 cc of alcohol, cork, shake well and often during one hour, and let stand over night. Transfer as much of the mixture as possible to a small percolator, the neck of which is provided with a purified cotton plug, receiving the percolate in a beaker or flask. Rinse the residue in the flask into the percolator with additional portions of the ether-chloroform mixture, packing it down moderately with a glass rod, and continue the percolation with the same mixture until the percolate does not give an alkaloidal reaction, or at most only an opalescence when 2 cc are evaporated, and the residue is treated with N/10 hydrochloric acid, the mixture filtered and the filtrate tested with Mayer's solution. Evaporate or distil the percolate to about 10 cc at a temperature not exceeding 70° C. Transfer to a separator, and rinse the vessel with two portions of 20 cc each of ether, alternating with two portions of 10 cc each of 2 per cent sulphuric acid. Shake out, making sure that the reaction is acid, and repeat the operation three times with 15 cc of the same strength of acid, filtering the acid solutions into another separator through a 7 cm filter, and rinsing the latter with a few cubic centimeters of water. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and if colored repeat until no more color is acquired. Make the solution alkaline with ammonia water and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(a) *Gravimetric*. Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(b) *Volumetric*. Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 3 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

Aliquot method.

Into a 200 cc flask weigh 15 grams of the powdered drug, add 150 cc of ether-chloroform mixture (5 to 1 by volume), cork and shake often for several minutes. Add 10 cc of ammonia water (10 per cent), shake frequently during one hour, and let stand over night. Add 15 cc of water, or sufficient to agglomerate the drug, shake, let settle a few minutes, and then decant 75 cc of the clear solution into a graduated cylinder. Transfer the solution to a separator, rinsing the cylinder with a few cubic centimeters of ether-chloroform, and shake out with 20 cc (or sufficient to render acid) of 2 per cent sulphuric acid, repeating the operation three times with 15 cc of the same strength acid, and collect the acid solutions in another separator. Wash the acid solution with 10 cc of ether-chloroform mixture (1 to 3 by volume), discarding the latter, and, if colored, repeat until no more color is acquired. Make the solution alkaline with ammonia water, and shake out with four successive portions of about 20 cc each of ether-chloroform mixture (1 to 3); collect the latter in a separator, wash with 5 cc of water, transfer the ether-chloroform mixture to a tared flask or beaker, rinsing the separator with a small portion of ether-chloroform, and evaporate the solvent at a temperature not exceeding 70° C.

(a) *Gravimetric*. Add 3 cc of ether, evaporate and dry to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

(b) *Volumetric*. Dissolve the residue in about 10 cc of neutral alcohol, add 25 cc of water, 5 to 15 drops of cochineal indicator solution, measure in from a burette a slight excess of N/50 sulphuric acid, mix intimately, and titrate back with N/50 potassium hydroxid. Report net amount of N/50 acid required.

Whenever iodeosin indicator is used, follow the directions of the United States Pharmacopœia VIII, page 542. Solutions standardized by means of cochineal should not be used with iodeosin without running a blank for comparison, as the indicators do not give the same neutral point.

These methods and those referred to in the United States Pharmacopœia are applied to the analysis of the following drugs with the modifications noted.

ACONITE ROOT.

Method I.

U. S. Pharmacopœia VIII, p. 28. Report the amount of N/10 acid required.

Method II.

Aliquot gravimetric. Use *ether* instead of ether-chloroform mixture in the final shaking out.

BELLADONNA LEAVES.

Method I.

U. S. Pharmacopœia VIII, p. 67. Report indicator used and amount of N/10 acid required.

Method II.

Aliquot volumetric.

BELLADONNA ROOT.

Method I.

U. S. Pharmacopœia VIII, p. 68. Report indicator used and amount of N/10 acid required.

Method II.

Aliquot volumetric.

CINCHONA BARK.

Method I.

U. S. Pharmacopœia VIII, p. 102. Report total and ether-soluble alkaloids.

Method II.

Total extraction, gravimetric.

COCA LEAVES.

Method I.

U. S. Pharmacopœia VIII, p. 106. Report indicator used and amount of N/10 acid required.

Method II.

Aliquot gravimetric. Use *ether* instead of ether-chloroform mixture throughout.

COLCHICUM CORM.

Method I.

U. S. Pharmacopœia VIII, p. 111. Report weight of alkaloidal residue.

Method II.

Exhaust 10 grams of the powdered drug by percolation with alcohol, add 25 cc of water to the solution and evaporate. Digest and stir the residue for several minutes with 25 cc of petroleum ether and transfer the liquid to a beaker, rinsing the residue with a little fresh petroleum ether. Add to the beaker 20 cc of water and evaporate the petroleum ether. Gently warm the first residue with 10 cc of water, cool, and filter the solution through a small filter into a separator, rinsing the vessel, and filter with several small portions of water and finally with the water in the beaker. Shake out the liquid with four portions of 10 cc each of chloroform and evaporate the latter in a tared dish. Add 3 cc of alcohol and evaporate, repeat the operation and dry the residue to constant weight at a temperature not exceeding 70° C. Report weight of alkaloidal residue.

COLCHICUM SEED.

Method I.

U. S. Pharmacopœia VIII, p. 112. Report weight of alkaloidal residue.

Method II.

See Colchicum Corm. Method II.

STANDARDIZATION OF VOLUMETRIC SOLUTIONS.

In order to compare the present pharmacopœial method for preparing volumetric solutions, a quantity of potassium bitartrate was purified according to the prescribed directions of the Pharmacopœia. Two equal parts of this purified potassium bitartrate were weighed out, one portion ignited at a low red temper-

ature in a platinum dish, dissolved in water, and carefully mixed with the second portion weighed as above. The resulting mixture was acid in reaction to phenolphthalein, indicating that some of the bitartrate was possibly lost during the ignition. Several repetitions of the above indicated that there apparently was some loss on ignition and that the method indicated in the Pharmacopœia required further study. Recourse was then had to normal sulphuric acid prepared by the usual methods, titrated by two independent workers using several indicators, with satisfactory results, the variations ranging from the lowest for methyl orange, 0.9890, to the highest for phenolphthalein, 1.0065. This solution was used as the basis for preparing decinormal sulphuric acid.

Mr. Parker's portion of the work was practically completed when the pharmacopœial corrections appeared, and his results are therefore based upon the original methods, except that cochineal had been substituted for hematoxylin. The standard acid employed by him, however, was found to correspond closely with that employed by the other analysts.

The results obtained are given in the following table:

*Comparison of methods for the assaying of aconite, belladonna, cinchona, coca leaves, and colchicum.**

Analyst.	Aconite leaves.			Aconite root.		
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II, aliquot.	
		Gravi- metric.	Volu- metric.		Gravi- metric.	Volu- metric.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Parker.....	0.243	0.241	0.179	0.486	0.588	0.410
Do.....	.256	.245	.154	.499	.447	.384
Do.....						
Rieger.....	.173	a.234	a.173	.499	.517	
Do.....	.173			.416	.528	
Do.....					.516	
Do.....						
Warren.....	.167	.231		.335	.612	.610
Do.....	.172	.254		.346	.611	
Do.....	.157					
Average.....	.191	.241	.169	.430	.546	.468
Maximum.....	.256	.254	.179	.499	.612	.610
Minimum.....	.157	.231	.154	.335	.447	.384
Difference.....	.099	.023	.025	.164	.165	.226

Analyst.	Belladonna leaves.			Belladonna root.		
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II, aliquot.	
		Gravi- metric.	Volu- metric.		Gravi- metric.	Volu- metric.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Parker.....	0.344	0.362	0.229	0.494	0.530	0.384
Do.....	.356	.409	.224	.488	.516	.384
Do.....	.356					
Rieger.....	.287		.253	.406		.423
Do.....	.244		.240	.370		
Do.....	.270			.407		
Do.....	.270					
Warren.....	.242		.264	.466		.403
Do.....	.284		.254	.460		.387
Do.....						
Average.....	.295	.386	.244	.442	.523	.396
Maximum.....	.356	.409	.264	.494	.530	.423
Minimum.....	.242	.362	.224	.370	.516	.384
Difference.....	.114	.047	.040	.124	.014	.039

* Mean of two assays.

Comparison of methods for the assaying of aconite, belladonna, etc.—Continued.

Analyst.	Cinchona (yellow).					Cinchona (red).				
	Method I, U. S. P., alkalo- loid.		Method II, total ex- traction alkaloid.			Method I, U. S. P., alkalo- loid.		Method II, total ex- traction alkaloid.		
	Total.	Ether soluble.	Total.	Ether soluble.	Marc.	Total.	Ether soluble.	Total.	Ether soluble.	Marc.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Parker	8.11	6.72	8.52		0.26	5.90	4.10	5.06		
Do	8.02	6.55	8.41		.08	5.27	4.27	4.91		
Do	8.24	6.81	8.08		.59					
Rieger	7.85	6.07	7.11	4.45	.30	5.64	3.64	5.40	3.05	0.31
Do	8.20	4.93	6.85	4.81	.49	5.73	3.25	5.32	3.01	.18
Warren	8.52	5.83	8.26	5.37	.40	5.48	3.51	5.38	3.41	.59
Do	8.75	4.98	7.53	5.09	.43	5.56	2.86	5.68	3.46	.35
Do	8.79	5.34	7.03	4.67	.28					
Average	8.31	5.90	7.72	4.88	.35	5.59	3.61	5.29	3.23	.36
Maximum	8.79	6.81	8.52	5.37	.59	5.90	4.27	5.68	3.46	.59
Minimum	7.85	4.93	6.85	4.45	.08	5.27	2.86	4.91	3.01	.18
Difference	.94	1.88	1.67	.92	.51	.63	1.41	.77	.45	.41

Analyst.	Coca leaves.			Colchicum corm.		Colchicum seeds.	
	Method I, U. S. P.	Method II, aliquot.		Method I, U. S. P.	Method II.	Method I, U. S. P.	Method II.
		Gravi- metric.	Volu- metric.				
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Parker				0.454	0.275	0.940	0.611
Do	0.498	0.738	0.558	.39	.283	.940	.597
Do	.510	.708	.540	a (.39)		a (.392)	
Rieger				.354	.282	.846	.496
Do	.613	.724	.592	.334	.260	b (.532)	b (.411)
Do	.569	.700	.626			1.044	1.190
Do						b (.526)	b (.452)
Warren	.382	.643	.400	.316	.286	.642	.658
Do	.407	.592	.348	.290	.298	.722	.588
Average	.496	.684	.511	.356	.281	.856	.690
Maximum	.613	.738	.626	.454	.298	1.044	1.190
Minimum	.382	.592	.348	.290	.260	.642	.496
Difference	.231	.146	.278	.164	.038	.402	.694

^a Residue purified by re-resolution in ether, treatment with water, etc., as in the assay of colchicum corm, U. S. P., not included in average, maximum, or minimum.

^b Residue purified as above, substituting petroleum ether for ether, not included in average, maximum, or minimum.

DISCUSSION OF RESULTS.

ACONITE LEAVES.

The leaves were assayed by the same methods as the root. The variation by the United States Pharmacopœia method reached 51 per cent of the average value. The aliquot gravimetric method gave more uniform results, the variation amounting to 10 per cent; but titration in a few cases indicated that, as is frequently the case, the gravimetric results are high. The Squibb physiological test was obtained with a dilution of 1-80. It will be noted that the indications by this test and by assay do not correspond to the analogous results for aconite root.

ACONITE ROOT.

For aconite root, as for the leaves, the aliquot gravimetric results showed less variation than the United States Pharmacopœia method, but the few titration results indicate that some of the former are considerably too high. The

Squibb physiological test gave a value of 1-500, or six times as great as the leaf, instead of two to three times, as might be inferred from the assays.

BELLADONNA LEAVES.

The results of Mr. Parker by the United States Pharmacopœia method for belladonna leaves are decidedly higher than those of the other analysts, which do not vary much among themselves. The discrepancy may indicate some deterioration in the drug in the three months intervening before the lower results were obtained. All the aliquot volumetric results, which were obtained about the same time, are fairly concordant. Experiments were made to determine whether, in the extraction of the drug by ethereal solvents, the presence of a small amount of water exercises an unfavorable influence. In the pharmacopœial method a few cubic centimeters are introduced in the form of diluted ammonia water. Mr. Parker varied the method by using alcoholic ammonia of corresponding strength (about 4 per cent), also by diluting stronger ammonia water with alcohol instead of water to the same strength. The results were decidedly lower, namely, 0.298 and 0.276 per cent.

Mr. Warren employed the total extraction method also, which is similar to the pharmacopœial method, except in the use of alcohol for diluting the ammonia. He obtained higher gravimetric results, but titration of the residues indicated that the increase was due to impurities.

Mr. Rieger tried the total extraction method, using alcohol in one test and water in another, to dilute the ammonia water. His results are low in both cases, and no conclusions can be drawn except that the residues are impure.

BELLADONNA ROOT.

In the results for belladonna root, as in those for the leaves, the best agreement is obtained by the aliquot volumetric method, and the gravimetric results are evidently vitiated by impurities. Just as with belladonna leaves, the results of experiments on the influence of small amounts of water on the extraction of the root led to no conclusion. No positive advantage was shown to accrue from the substitution of alcohol.

CINCHONA (YELLOW AND RED).

The pharmacopœial method for cinchona gave much less variation in the results on total alkaloid than the total extraction method, but, as was the experience last year, the results on ether-soluble alkaloid are not concordant.

The change in the pharmacopœial method, doubling the amount of solvent used in extracting the drug, involves the shaking out of 200 cc of ether-chloroform mixture with comparatively small volumes (15, 10, and 5 cc) of dilute acid, which is a tedious and difficult task. Evidently some nonalkaloidal matter passes into the acid solution in shaking out, and from that into the alkaloidal residue, which is discolored in all cases.

In determining ether-soluble alkaloid Mr. Warren filtered the ethereal solution through cotton in a small funnel.

The total extraction method fails to exhaust the marc even after obtaining 400 to 500 cc of percolate, though weak runnings give no test for alkaloids. On testing the marc, as directed last year, by boiling with normal hydrochloric acid and shaking out the acid extract, the amounts of impure alkaloid indicated in the column under "marc" were obtained.

COCA LEAVES.

The results by all methods for coca leaves are so variable that none can be said to be encouraging. The results of the aliquot gravimetric method agree best among themselves, but the titration equivalents show that they must be regarded as too high.

COLCHICUM CORM.

The results for colchicum corm by the pharmacopœial method are materially higher than by Method II, but also vary relatively more among themselves.

COLCHICUM SEEDS.

Results for colchicum seeds, by both methods, exhibit great variations, and experiments on purifying the residues by a process similar to the latter part of the pharmacopœial method for colchicum corm showed that the impurities present would render the results entirely unreliable unless they were removed.

In the absence of the referee on tannin, his report was presented by Mr. Tolman, as follows:

REPORT ON TANNIN.

By F. P. VEITCH, *Referee*.

The referee was unable to obtain the samples desired until very late, and for this reason very few of the twelve or fifteen analysts, who had promised to collaborate, were able to do so and the work was confined to a comprehensive study of the filtration of soluble solids through folded S and S 590 and S and S 588 paper, the first a high-grade paper and the latter a cheaper paper of ordinary grade. For a number of years the No. 590 paper has been unsatisfactory for use in soluble-solids determinations, as it is not uniform and is too weak to stand the weight of the solution after the paper is folded.

In a study which the referee had made, using a large number of different extracts, it was found that the No. 588 paper gave results agreeing closely with those with the No. 590 paper, except in the case of quebracho. Reed^a had previously obtained satisfactory results with No. 588, but only on hemlock and lentiscus. The referee's favorable results led him to submit the paper to collaborative tests, as both time and expense would be saved if it could be used. The following circular letter was sent out:

DIRECTIONS FOR TANNIN WORK, 1907.

DEAR SIR: I am forwarding you to-day three samples of extracts in which the following determinations are to be made in triplicate:

- (1) Total solids.
- (2) Soluble solids, using 15 cm 590 S and S folded filters.
- (3) Soluble solids, using 15 cm 588 S and S folded filters.

Make all dryings on the bottom shelf of a boiling-water oven, drying for 12 hours.

Dissolve the following quantities of extracts in 2,700 cc of water at 80° C: Sample No. 1, 18 grams; Sample No. 2, 42 grams; Sample No. 3, 15 grams.

Allow to cool overnight to 20° and dilute to 3,000 cc. In these solutions determine total and soluble solids as follows:

Total solids: Thoroughly mix the solution, immediately pipette 100 cc into a tared dish, evaporate, and dry as directed.

^a J. Soc. Chem. Ind., 1902, 21: 691.

Soluble solids: Add 75 cc of solution (kept at from 20° to 25° C during filtration) to 2 grams of kaolin (free from soluble salts); stir, let stand 15 minutes, decant, and discard as much as possible of the supernatant liquid. Again add 75 cc of the tannin solution to the kaolin, stir, and pour immediately on a folded filter. Keep the filter full and the funnel and receiving vessel covered. Reject the first 150 cc of filtrate, evaporate and dry the next 100 cc (which must be as clear as practicable) as directed. * * *

If you have not the kind and size of filter paper specified please communicate with the referee before proceeding further.

The results reported are given in the following table:

TABLE I.—*Determinations of total and soluble solids in three extracts, comparing S and S 588 and 590 filter papers.*

Analyst.	Temperature.	Total solids.			Soluble solids.			
		Sample 1.	Sample 2.	Sample 3.	Sample 1.			
					S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a
	° C.	Per cent.	Per cent.	Per cent.	Per cent.	Minutes.	Per cent.	Minutes.
F. C. Sammett.....	25	85.8	45.9	89.5	80.0	32-42	79.6	35-45
C. C. Smoot.....	26	84.4	45.2	88.3	78.7	54-81	78.5	56-85
Do.....	20	84.8	45.5	88.7	77.8	52-82	77.6	55-80
F. P. Veitch.....	24	85.1	45.5	88.8	80.2	95-140	79.3	90-110
Chas. Eachus.....		84.4	45.2	88.2	78.3		76.3	
A. G. Stillwell.....		87.7	45.3	90.6	82.5		79.8	

Analyst.	Temperature.	Soluble solids.							
		Sample 2.				Sample 3.			
		S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a	S and S 590 filter paper.	Time. ^a	S and S 588 filter paper.	Time. ^a
	° C.	Per ct.	Min.	Per ct.	Min.	Per ct.	Min.	Per ct.	Min.
F. C. Sammett.....	25	44.6	34-38	44.5	37-40	88.3	35-30	87.8	33-25
C. C. Smoot.....	26	44.4	33-39	44.2	30-35	87.8	21-23	87.4	25-23
Do.....	20	42.0	70-150	42.5	54-115	87.9	27-23	87.7	30-24
F. P. Veitch.....	24	44.4	48-52	44.3	46-45	87.9	39-31	87.5	32-22
Chas. Eachus.....		44.2		43.6		87.2		86.8	
A. G. Stillwell.....		43.6		43.3		89.2		88.6	

^a Under "Time" the first figures show the time required for the first 150 cc; the second figures for the next 100 cc.

No final conclusion can be drawn from these few results, but it is quite evident that the S and S 588 paper gives slightly lower results than the 590 paper under the conditions of filtration, which, it must be remembered, were devised for use with the 590 paper. The results agree in general with those that have been previously obtained, and it seems probable that with some modification in the method of filtration, such as the filtration of about 200 cc before collecting the portion for analysis, the results would agree with those obtained with the 590 paper. Such a procedure would, of course, lengthen the time of filtration, and this would not be desirable.

The time of filtration with the 588 paper was, as a rule, slightly shorter than with the 590 paper. Mr. Smoot observed that the temperature at which filtration was conducted affected the speed of filtration as well as the percentage of soluble solids; the cooler the solution the more slowly it filtered. Mr. Smoot also made some determinations with an asbestos-kaolin mat on a Buckner fun-

nel, using suction as proposed by Reed at the recent meeting of the Association of American Leather Chemists. The results were as follows:

TABLE II.—*Determination of soluble solids in tannin extracts using an asbestos-kaolin mat, at 21° C.*

Sample 1.		Sample 2.		Sample 3.	
<i>Per cent.</i>	<i>Minutes.</i>	<i>Per cent.</i>	<i>Minutes.</i>	<i>Per cent.</i>	<i>Minutes.</i>
77.2	11-26	41.7	11-48	87.8	4-8
77.4	12-28				
78.1	15-45	41.6	20-52	88.4	4-3
77.4	20-68				
78.6	20-68	41.7	21-62		

The first figure under "Minutes" shows the time required for the first 150 cc; the second figure for the next 100 cc, with the exception of the first two results on Sample 1 and both results for Sample 3 which were obtained after filtering only 100 cc in each case.

As will be seen, the time required for filtration when the asbestos mat was used was greatly reduced. When the quantity of liquor rejected was at least 150 cc, the results agreed fairly well with those obtained with S and S 590 paper, except on Sample 2 which gave exceptionally clear filtrates with the asbestos filter. This may account for the low results.

The referee does not recommend a change of papers for filtering soluble solids, as the results obtained with 588 paper, while usually sufficiently close to those by the 590 paper, are apt to be wide on quebracho. Doubtless the 588 paper could be safely used on tannery liquors and other materials of low tannin content.

RECOMMENDATIONS.

It is recommended—

(1) That the work on the filtration of tannin extracts be continued with a view to finding a cheap, strong paper as a substitute for S and S 590.

(2) That the method of filtering soluble solids suggested by Reed be given further trial.

(3) That the referee take up the study of methods for the analysis of leather, as such methods are greatly needed.

REPORT ON SOILS.

By J. H. PETTIT, *Referee.*

The work of the referee on soils for the present year has been mainly a continuation of that undertaken last year. Two of the four samples used were the same as two of those sent out last year, while Samples 1 and 3 were new. The following outline of the work to be done was sent to those desiring to participate:

(a) Determine dry matter upon 5 grams of the air-dried soil by heating five hours in water oven.

(b) Make a preliminary digestion of the air-dried soil in fifth-normal nitric acid at room temperature.^a Weigh 200 grams of soil into a 2½ liter

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 46, Revised, p. 74 (b) ; Bul. 107, p. 18 (b).

flask and add 2,000 cc of a solution of nitric acid of such strength that, after allowing for the nitric acid neutralized by the soil, as shown by the previous digestion, there will be left 2,000 cc of fifth-normal nitric acid solution. Digest five hours at room temperature, shaking every half hour. Record the temperature of digestion. At the end of the period shake and filter through a large folded filter, pouring the solution back into the filter until it runs through clear.

Evaporate 1,000 cc of the filtrate to dryness, moisten with hydrochloric acid, dry again, heat one hour at 110° C to insure complete removal of acid, take up with hydrochloric acid and water, heating if necessary, filter out silica, and wash. Evaporate filtrate and washings to about 10 cc. Add 5 cc of concentrated nitric acid, neutralize with ammonia, make barely acid with nitric acid, heat to from 40° to 50° C, precipitate with ammonium molybdate solution,^a and let stand over night. Filter, wash with 0.1 per cent ammonium nitrate solution until free of acid, and twice with cold water. Dissolve in a standard potassium hydroxid solution, 1 cc of which contains 8.324 mg of potassium hydroxid and is equivalent to 0.2 mg of phosphorus. Titrate the excess with a nitric acid solution, 1 cc of which is equivalent to 1 cc of the potassium hydroxid solution.

(c) Weigh 10 grams of sodium peroxid into an iron or porcelain crucible and thoroughly mix with it 5 grams of soil; if the soil is very low in organic matter add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly upon the surface of the charge and the sides of the crucible until the action starts. Cover crucible until reaction is over, and keep at a low red heat for twenty-five minutes; do not allow fusion to take place. By means of a large funnel and a stream of hot water transfer the charge to a 500 cc measuring flask. Acidify with hydrochloric acid and boil; let cool and make up to the mark. If the action has taken place properly there will be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc of the clear solution.

Precipitate the iron, aluminum, and phosphorus from the hot solution with ammonium hydroxid; do not allow to settle; filter; wash three or four times with hot water; return the precipitate to the beaker with a stream of water, holding the funnel over the beaker, and dissolve the precipitate in hot dilute hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remaining. Remove silica and determine phosphorus as in (b). Determine phosphorus, using the alkali carbonate fusion method^b and getting rid of the silica as suggested above.

(d) Fuse 1 gram of soil according to the well-known J. Lawrence Smith method. Transfer the fused mass to a porcelain dish, slake with hot water, grind finely with an agate pestle, and transfer to a filter. After washing free of chlorids, concentrate the filtrate and washings in a Jena beaker to about 20 cc and filter. Slightly acidify filtrate and washings with hydrochloric acid, concentrate in a platinum dish, and add $1\frac{1}{2}$ cc of a platinic chlorid solution (10 cc contains 1 gram of platinum). Evaporate to a sirupy consistency as usual and wash with 80 per cent alcohol and an ammonium chlorid solution. Determine potassium according to the regular J. Lawrence Smith method.^c

The following laboratories sent reports:

S. D. Averitt, for the Kentucky Experiment Station.

H. D. Edmond, for the Connecticut Experiment Station.

R. W. Thatcher, for the Washington Experiment Station.

G. S. Fraps, for the Texas Experiment Station.

J. H. Norton, for the Arkansas Experiment Station.

J. H. Pettit, for the Illinois Experiment Station.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 46, Revised, p. 11 (b); Bul. 107, p. 2 (c).

^b Fresenius's Quantitative Chemical Analysis, p. 422.

^c Ibid, p. 426.

TABLE I.—*Determination of phosphorus soluble in fifth-normal nitric acid.*

[Parts per million in dry soil.]

Analyst.	Temperature of extraction.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
	°C.				
S. D. Averitt, Kentucky.....	29	{ 15.4 11.4 }	-----	187.4	{ 185.0 187.0 }
H. D. Edmond, Connecticut ^a	25	10.3	8.2	182.6	188.2
H. R. Watkins, Washington.....		8.8	{ 7.3 7.5 }	195.0 167.2	167.2 177.0
J. H. Norton, Arkansas.....		{ 14.9 15.1 }	12.7	{ 193.8 189.4 }	169.0 175.2
A. Ystgard, Illinois.....	25	{ 12.7 12.7 }	10.0 10.0	168.3 169.0	167.0 166.2
S. E. Asbury, Texas ^a	30	10.4	-----	157.2	-----
E. Carlyle, Texas ^a	30	11.0	-----	-----	-----

^a Reported by analysts as phosphoric acid and calculated to phosphorus; duplicates not given.TABLE II.—*Determination of phosphorus by the sodium peroxid fusion.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky.....	{ 0.043 .041 }	-----	0.095 .101	0.103 .099
H. D. Edmond, Connecticut ^a	.045	0.055	.116	.101
J. H. Norton, Arkansas.....	{ .046 .044 }	.065 .059	.108 .108	.108 .094
A. Ystgard, Illinois.....	{ .047 .046 }	.051 .052	.100 .102	.100 .100
A. W. Gregory, Illinois.....	{ .040 .040 }	.046 .046	.096 .094	.084 .082
J. H. Pettit, Illinois.....	-----	-----	{ .096 .094 }	.090 .088

^a Duplicates not reported.TABLE III.—*Determination of phosphorus by alkali carbonate fusion.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky ^a	0.035	-----	0.092	0.097
A. Ystgard, Illinois.....	{ .046 .046 }	0.052 .052	.103 .105	.100 .100

^a Duplicates not reported.

It is unfortunate that no more work by the standard method was done. The results obtained by the sodium peroxid method show fair agreement with the meager data obtained by the standard alkali carbonate method.

TABLE IV.—*Determination of total potassium by the regular method.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
H. D. Edmond, Connecticut ^a	0.461	0.590	0.477	0.572
H. R. Watkins, Washington.....	{ 1.227 1.381 }	1.790 1.620	2.449 2.395	1.732 1.719
A. Ystgard, Illinois.....	{ 1.453 1.438 }	1.852 1.838	1.771 1.765	1.826 1.808

^a Reported unsatisfactory, but no time to repeat.

TABLE V.—*Determination of total potassium by modified method.*

[Percentage of dry soil.]

Analyst.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.
S. D. Averitt, Kentucky a.....	1.250		1.270	1.485
A. Ystgard, Illinois.....	1.449	1.846	1.789	1.818
	1.447	1.843	1.778	1.793

^a Duplicates not reported.

RECOMMENDATIONS.

It is recommended that—

- (1) The modified J. L. Smith method for total potassium be further tested.
- (2) The sodium peroxid fusion method for total phosphorus be adopted as a provisional method of this association and be further tested.
- (3) The "Determination of Volatile Matter" (U. S. Dept. Agr., Bureau of Chemistry, Bul. 46, Revised, p. 72; Bul. 107, p. 14) be replaced by the "Determination of Total Organic Carbon" (J. Amer. Chem. Soc., 1904, 26: 1640).

REPORT ON INORGANIC PLANT CONSTITUENTS.

By W. W. SKINNER, *Referee.*

The work upon inorganic plant constituents has been a continuation of the scheme outlined for the preceding year. The chemists of six experiment stations signified their willingness to undertake the cooperative work, and samples were sent to each, but only a partial report from one station has been received. The lack of interest shown in the work has been rather discouraging to the referee, and it would seem that the objects of the association are defeated unless more cooperative work on proposed methods can be obtained.

The table given below is a tabulated statement of the sulphur content of two samples of cotton-seed meal and two samples of mustard-seed meal determined by three methods. The results are expressed on a moisture-free basis, and the sulphur calculated to sulphanion (SO_4). For purposes of comparison the sulphur content of the samples used the previous year are included.

TABLE I.—*Determination of sulphur as SO_4 in moisture-free cottonseed meal.*

Analyst.	Sample A.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. W. Skinner.....	{ 0.02	1.62	1.64	1.73	0.20	
	{ .05	1.56	1.61	1.74	.17	
Average.....			1.63	1.74	.19	.89
C. E. Goodrich.....				{ 1.69	.16	
				{ 1.67	.13	
Average.....				1.68	.15	.91

TABLE I.—*Determination of sulphur as SO₄ in moisture-free cottonseed meal—Continued.*

Analyst.	Sample C.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. W. Skinner.....	{ 0.17	1.57	1.74	1.62	0.20	
					.16	
Average.....				1.62	.18	.89
E. I. Lichti.....	.43	1.78	2.21	{ 1.66	.16	
				1.64	.16	
Average.....				1.65	.16	.90
O. M. Shedd.....				{ 1.51		
				1.58		
Average.....				1.55		
Geo. Roberts.....				{ 1.65		
				1.61		
Average.....				1.63		

TABLE II.—*Determination of sulphur as SO₄ in moisture-free mustardseed meal.*

Analyst.	Sample B.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. W. Skinner.....	{ 0.13	4.11	4.24	4.37	0.40	
	.21	4.15	4.26	4.43	.33	
Average.....			4.25	4.40	.37	.92
C. E. Goodrich.....				{ 4.33	.39	
				4.34	.30	
Average.....				4.34	.35	.92

Analyst.	Sample D.					
	Method employed.					
	Combustion.			Peroxid.	Ordinary ashing.	
	Nonvol- atile.	Volatile.	Total.	Total.	Total.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. W. Skinner.....	0.40	3.67	4.07	4.18	0.46	0.89
E. I. Lichti.....				{ 4.56	.47	
				4.42	.50	
Average.....				4.49	.49	.89
O. M. Shedd.....				{ 4.44		
				4.57		
Average.....				4.51		

The results for total sulphur by the peroxid method are fairly satisfactory and the referee recommends that this method, which is now provisional, and which was recommended last year for adoption, be made an official method.

Two of the analysts reported considerable difficulty with the peroxid method, especially with mustard-seed meal. This was probably due to one of two causes. Either the charge was not thoroughly moistened before the addition of the sodium carbonate and sodium peroxid, or it was not allowed to stand a sufficient length of time after the addition of the peroxid for the preliminary oxidation to occur. If this oxidation is not completed before the charge is placed over the flame, almost invariably the contents will be blown from the crucible by the violent action when it does begin.

The results by the combustion method are interesting and significant. The results for total sulphur, calculated to sulphuric acid (SO_4) agree within 0.1 per cent with the results by the peroxid method, while the results upon nonvolatile sulphur by this method are in striking agreement with the sulphur obtained in the ash by the ordinary method of ashing.

The combustion method is probably too slow and the technique too intricate for it to become the routine method for ash analyses. As an exact method for determining separately the volatile and nonvolatile mineral constituents of organic materials, it is, however, of great importance, and is therefore recommended for provisional adoption. The percentage loss of sulphur by the ordinary ashing method is shown in the table. On sample A, cotton-seed meal, the loss found by two analysts was, respectively, 89 per cent and 91 per cent, and on sample D, 89 per cent and 90 per cent. On samples C and D, mustard-seed meal, the loss was 92 per cent and 92 per cent, and 89 per cent and 89 per cent by two analysts.

The loss of sulphur by volatilization is very important in the interpretation of those proximate analyses in which ash is one of the factors. Thus in the analysis of a cattle food the determination of ash, by the ordinary method of incineration in air, constitutes one of the separations, the percentage of moisture, ash, fat, protein, and crude fiber being added together and subtracted from 100 to obtain the percentage of nitrogen-free extract. It is evident, therefore, that in a product with a sulphur content similar to mustard-seed meal, in which the loss of sulphur is about 4 per cent, the nitrogen-free extract would be just so much too high, provided the sulphur was not accounted for in some other form. The matter is further complicated by the fact that the sulphur is partially accounted for in the protein, as the protein figure is obtained by multiplying the nitrogen content by a factor which includes sulphur in the formula for the protein molecule.

When the association decided at the meeting in 1901 to adopt the name "inorganic plant constituents" to replace "ash," by inference, at least, the older methods for ash were abandoned. The referee believes this was a mistake which has resulted in considerable confusion and to which attention was specifically called in his report for last year. At present the official method is by ignition with calcium acetate, which in many cases is impracticable. For instance, in the analysis of a forage crop one of the determinations is ash, and it is frequently important to determine in the ash the mineral constituents. If, however, the mineral constituents in the material are determined by the calcium acetate method it is impossible to translate them in terms of the ash as determined by the official method for ash in feeds and feeding stuffs. The result has been that workers along this line have continued to use the methods for ash given in Bulletin 46. Revised, although now not official. It seems advisable, therefore, that this method of ashing by charring, extracting, etc., be

again inserted in our methods as an official method, and the referee recommends this.

There has never been an official method for the determination of iron and aluminum in ash. It is therefore recommended that the development of methods for this purpose be especially referred to the referee for next year.

RECOMMENDATIONS.

It is recommended that—

- (1) The peroxid method be adopted as the official method.
- (2) The combustion method be adopted as provisional.
- (3) The official method for ash given in Bulletin 46, page 77, be again adopted.
- (4) The development of methods for determining iron and aluminum in ash be referred to the succeeding referee.

THE PHOSPHORIC ACID OF THE SOIL.

By G. S. FRAPS.

It is the purpose of this paper to present briefly some of the more important results which have been obtained at the Texas experiment station in the study of the phosphoric acid of the soil. The work has been confined chiefly to a study of the phosphoric acid soluble in fifth-normal nitric acid, and in ammonia under the conditions of the humus extraction.

AMMONIA-SOLUBLE PHOSPHORIC ACID.

The solubility of the phosphoric acid of mineral phosphates in ammonia was first studied, and it was found that ferric phosphate, vivianite, and wavellite give up a large part of their phosphoric acid to ammonia, while the calcium phosphates do not. It thus appears probable that some of the phosphoric acid soluble with humus in ammonia is of inorganic origin.

Twelve soils were then treated with potassium phosphate and a comparison made of the phosphoric acid soluble with humus before and after the treatment. We found an increase in so-called humic phosphoric acid in some of the soils, which proves that the absorbed phosphoric acid is soluble in ammonia, and some of the phosphoric acid soluble with humus must be of mineral origin. One of these soils, which is a subsoil and contains practically no humus, was nearly as rich as some black Minnesota soils in ammonia-soluble phosphoric acid, showing 0.06 per cent before and 0.228 per cent after treatment.

The distribution of the phosphoric acid in the ammonia extract as prepared for the estimation of humus was also studied. About one-ninth of the phosphoric acid is precipitated with the clay and is probably held in mechanical suspension. About one-third is precipitated with the organic matter and is probably in combination with it. About four-ninths remains in solution and is probably of mineral origin. These figures represent average results from seven Minnesota soils rich in humus.

The phosphoric acid associated with the organic matter does not diffuse and is not increased when the organic matter is precipitated in the presence of phosphoric acid, nor decreased when the organic matter is dissolved in ammonia and reprecipitated. It is concluded that the phosphoric acid is in organic combination.

The organic phosphoric acid is present in soils in much smaller quantity than has generally been assumed, even in soils rich in humus. On account of the smallness of the quantity in the soil and the slowness of decomposition of the

humus, the organic phosphoric acid can not be considered as of great importance, except possibly in some soils rich in humus, where it may serve as a reserve store of phosphoric acid.

An increase in the phosphoric acid soluble in ammonia during processes of decay does not necessarily mean that phosphoric acid has entered into organic combination.

PHOSPHORIC ACID SOLUBLE IN FIFTH-NORMAL ACID.

Efforts were also made to ascertain what mineral phosphates are affected by the fifth-normal nitric acid. Two thousand cc of solvent and 0.2 gram of phosphoric acid mineral were brought together for five hours at 40° C. and phosphoric acid was determined in the filtrate. It was found that practically all of the phosphoric acid of iron, aluminum, and calcium phosphates, and the minerals phosphorite, vivianite, triplite, and apatite was dissolved. Wavellite, dufrenite, and variscite were only slightly affected. Experiments with other solvents gave similar results.

The conclusion drawn from this work is that the solvents can not differentiate phosphates of different agricultural value in the soil. To take an extreme case, equal quantities of phosphoric acid in acid phosphate and apatite would yield the same results with fifth-normal acid and other solvents, but when applied to the soil the crops produced would be decidedly different. We are therefore justified in comparing the phosphoric acid extracted by weak solvents only with soils which probably contain the same phosphates.

The next point considered was whether the phosphoric acid dissolved from the mineral phosphates was removed from solution by the soil; that is, if any fixation from the fifth-normal nitric acid occurred. This question was studied by ascertaining the amount of phosphoric acid dissolved, first, from the soil alone, and second, from the soil plus a known quantity of phosphate. It appeared that a number of soils have a high absorptive power for phosphoric acid from solution in fifth-normal nitric acid. There were at least three soils in which over 70 per cent of the added phosphoric acid (210 parts per million) were absorbed. Similar results were observed with other solvents tested. It is evident that the phosphoric acid extracted from some soils may represent less than 30 per cent of the phosphoric acid which actually went into solution. Two soils with the same amount of phosphoric acid extracted by fifth-normal nitric acid may really contain quite different quantities of soluble phosphates.

This question of absorption must be seriously considered in connection with soil analysis. Its significance and the correction that should be made for it are being studied at the Texas Station. We are satisfied that the fifth-normal nitric acid method will yield results of great practical value. A large number of pot experiments have been conducted, but chemical analyses can now take the place of such investigations to some extent, and we expect to discontinue pot experiments upon certain types of soils containing less than 20 parts per million of phosphoric acid, as all of these soils appear to be seriously deficient in phosphoric acid. Full details of this work will be published by the Texas station at a later date.

REPORT OF COMMITTEE ON NOMINATIONS.

Mr. B. L. Hartwell, chairman of the committee on nominations, submitted the following report: For president, Mr. Harry Snyder, of Minnesota; vice-president, Mr. W. D. Bigelow, Washington, D. C.; secretary, Mr. H. W. Wiley, Washington, D. C.

For additional members of the executive committee, Mr. B. B. Ross, Alabama, and Mr. G. F. Fraps, Texas.

On motion by Mr. Frear, the secretary was instructed to cast a unanimous vote for the officers nominated.

On behalf of the committee on food standards, Mr. Frear submitted a report on the progress made and the work done jointly with the committee appointed by the National Association of State Dairy and Food Departments.

REPORT OF COMMITTEE ON FERTILIZER LEGISLATION.

During the last few years the committee on fertilizer legislation has considered the subject in various aspects. First: We have studied the problem of whether or not national legislation respecting interstate trade in fertilizers is desirable. Without question national legislation respecting the use of fertilizers in the District of Columbia is extremely desirable, and likewise in those Territories which have not legislated on the subject themselves. At the present there is no law whatever controlling commerce in fertilizers in the District of Columbia. This, however, as can well be seen, is not a matter of very great importance, because the tilling of the soil is not the principal industry of the District of Columbia. It is doubtful, therefore, if it would be advisable for Congress to legislate upon this subject solely for the District.

Second: This committee has also been in correspondence from time to time with the manufacturers of fertilizers. It is our opinion that national legislation would be highly beneficial to the manufacturers of fertilizers provided a system of tagging and a method of expression of analysis could be adopted which would be satisfactory to practically all the States, in which case one label would be legal throughout the country. Attention should be called in this connection to the effect of the national food and drugs act in bringing into harmony the definitions of adulteration and misbranding in the various States. Already from 15 to 20 States have adopted the definitions of the food and drugs act in respect of adulteration and misbranding, either by direct legislative enactment or by the establishment of regulations by the food and drug authorities. It appears quite probable that this is only a beginning and that it is only a question of a few years when the definitions of adulteration and misbranding of foods and drugs will be practically the same in all the States. If this happy conclusion could be reached, it would be sufficient reason for actively favoring the passage of a national law controlling interstate commerce in fertilizers and fertilizing materials.

It is evident, however, that there would be many points of dispute, not only between the officials of the various States respecting the proper method of tagging and of expressing analytical results, but also a still wider disagreement between the State officials and the manufacturers. It seems, therefore, unwise to press the subject of national legislation in regard to fertilizers further until the officials of this association, representing as they do the majority of States exercising fertilizer control, can come to an agreement respecting a uniform system of labels and of methods of expressing analysis. This subject, as is well known, has been under discussion by our association for several years, and satisfactory and encouraging progress has been made. This leads to the hope that in a few years more the representatives of each State in this association may come to an agreement on this important subject.

Third: The committee does not deem it wise to favor any form of national legislation which would in any way interfere with the State system of control

of fertilizers. That is a matter with which, in our opinion, the National Government has nothing whatever to do. The systems of control, as is well known, are not uniform. In some States a tax is laid upon the gross tonnage of fertilizers sold. In other States a tax is laid upon the labels which are attached to the various packages, and other forms of control are exercised. It is believed that the wisest control of this kind is that which seems best to the State officials who have charge of such matters, and the State legislatures which establish the legal status of such control. This committee, therefore, is opposed to any national legislation which would attempt to influence the States in any way respecting the method of control of fertilizer sales within the States.

Fourth: After repeated attempts to secure suggestions from manufacturers, your committee is of the opinion that upon the whole the manufacturers themselves would rather not have national legislation and prefer to submit to the disadvantages of the present system rather than to see incorporated in a national law any system of stating analyses or the character of the materials used in the compounding of fertilizers which would be objectionable to them. This, however, does not seem a sufficient ground upon which to advise that no national action be taken, and it seems advisable to endeavor to secure from the manufacturers a full expression of their views in order that the matter may be widely discussed. If it were possible to extend the agreement among the State officials already referred to relating to methods of tagging and of stating the results of analytical work, so as to obtain the approval of the manufacturers, the difficulties in the way of a national law would be practically eliminated. In this case the enactment of such a law would prove beneficial to all parties by aiding in securing the agreement among the various States.

Fifth: Your committee therefore recommends that for the present no attempt be made to bring a national fertilizer law to the attention of the Congress with the object of controlling commerce in fertilizers in the District of Columbia and the Territories of the United States and in interstate commerce, but that, on the other hand, an effort be made to secure an agreement among all the State officials respecting the fundamental definitions of the misbranding and adulteration of a fertilizer, and a common understanding respecting the proper method of tagging or branding, and the proper method of stating the results of analysis; that an attempt be made to secure an agreement between the officials of the States and the fertilizer manufacturers respecting the proper method of referring to the crude sources of the plant food which may be present in any given fertilizer. This committee believes that all these points should be settled before any concerted effort is made to bring the matter of national fertilizer control to the attention of Congress. Further than this, your committee is of the opinion that when such an effort is made it should relate solely to the fundamental conditions above mentioned, and should be so conducted as not in any way to affect the States in respect to the proper methods of raising revenue from fertilizers sold under the State control.

Sixth: The committee is aware that this report is not fully in harmony with the last recommendation of the committee published on page 175 of Bulletin No. 105 of the Bureau of Chemistry. It is there recommended that the committee be continued, with instructions to secure, if possible, collaboration of the great fertilizing interests in the perfection of a measure which in the future may be submitted to Congress with the joint approval of the manufacturing interests and of the Association of Official Agricultural Chemists. It is hoped that such a measure may be perfected, but precedent to that it seems wise that the points mentioned above should be first considered.

Seventh: It is therefore recommended that the committee be continued with instructions to confer with any committee appointed by this association relating

to the methods of branding or tagging and the methods of expressing analysis, and that when agreement is secured in this association on these points the great fertilizing interests be again approached with a view to securing their adhesion to these points, and also to the definitions of adulteration and misbranding which may be adopted by this association. As a tentative basis of discussion, the accompanying definitions of misbranding and adulteration are submitted.

H. W. WILEY.

B. W. KILGORE.

H. B. McDONNELL.

B. B. ROSS.

J. L. HILLS.^a

TENTATIVE DEFINITIONS OF FERTILIZERS AND OF MISBRANDING AND ADULTERATION.

(1) A fertilizer shall be defined as any simple, compound, or mixed material, prepared for the purpose of selling, or sold, or offered for sale, to be applied to the soil as nourishment for plants, or as a modifier of the soil in any respect in its relation to the growth of plants. The term "fertilizer material" (or ingredients) shall include every plant-food material which is utilized, or intended to be utilized, in the manufacture, preparation, or mixing of the fertilizers defined above.

(2) A fertilizer, or fertilizer material (or ingredient), shall be deemed to be adulterated:

(a) If the percentage of any of its ingredients fall materially below the professed standard under which it is sold, whether this standard appear as a label upon the package or as a guaranty in any other way by the vendor thereof.

(b) If any of the ingredients thereof have an origin other than that indicated upon the package, or guaranteed in any other way by the vendor thereof.

(c) If any of the ingredients of the fertilizer, or fertilizer material, be in a state of combination different from that indicated by the label or guaranteed by the vendor thereof.

(3) A fertilizer, or fertilizer material, shall be deemed as misbranded:

(a) If any false name or misleading statement or design or device be affixed to any package thereof or used in any way as a representation of the materials thereof by the vendor.

(b) If any false or misleading statement respecting the origin of the material be made upon the label, or any statement or guaranty of the vendor.

(c) If any false or misleading statement be made upon the label, or by the vendor, respecting the country or origin of the materials of which the fertilizer is composed.

(d) If any false or misleading statement be made on the label, or by the vendor, respecting the virtues or qualities of the fertilizer or the materials composing it.

^a Mr. Hills, in signing the report, submitted certain criticisms and suggestions bearing on the tentative definitions, calling attention especially to the following points: (1) The danger of injustice to a product arising from imperfect mixing and mechanical separation in transit in the application of a percentage standard, as indicated in "2 (a)." (2) The difficulty of interpreting under "3 (a)" the words "misleading statements" or "design or device;" for example, in the application of the term "guano," or the labeling of fertilizers stated to be especially adapted for certain purposes, as, for example, "potato fertilizers," found to be low in potash, and therefore not especially applicable for this purpose.

(e) If sold under any false name or appellation, whether such name appear upon the package or label or be given to the article by the vendor thereof.

(f) If it be an imitation of or offered for sale under the name of another fertilizer or fertilizer material.

The report of the committee was adopted.

REPORT OF COMMITTEE ON THE TESTING OF CHEMICAL REAGENTS.

By L. F. KEBLER, *Chairman*.

During the past year little of importance has appeared in print dealing with chemical reagents. E. Merck's book on "Chemical Reagents, their Purity and Tests," which has been translated into English by Henry Schenck, contains several new features which are interesting, but a number of new titles for chemical reagents have been employed which can only lead to confusion.

The committee during the past year has been unable to devote any time to cooperative work on chemical reagents. The chairman has, however, partially completed an investigation of the quality and purity of the indicators now supplied to chemists, and thus far the results indicate that these materials are far from satisfactory. One indicator in particular, namely, pure litmus cubes, should be mentioned. Most of us have been under the impression that these cubes represented a highly purified litmus product, when as a matter of fact they consist of calcium carbonate pressed into cubes and impregnated with a solution of litmus, from 94 per cent to 96 per cent of calcium carbonate being present. It is the intention of the chairman to make a full report on indicators at the next meeting.

A large number of chemicals have been examined during the past year in the drug laboratory, with the result that there is a general improvement in the quality of the reagents supplied to the Bureau of Chemistry, but it frequently happens that chemicals sold as possessing the highest degree of purity are found to be unsatisfactory, being contaminated with foreign material which renders them unsatisfactory for analytical work. These data are being collected and will be prepared for publication.

It is recommended that the committee be continued for another year.

REPORT OF COMMITTEE ON UNIFICATION OF TERMS FOR REPORTING ANALYTICAL RESULTS.

R. J. DAVIDSON, *Chairman*.

This committee has endeavored for nearly three years to get an expression of opinion from the members of the association as to their views in regard to the nomenclature for reporting analyses of fertilizers, etc., but only comparatively few have responded. The committee decided at its last meeting that it would be best to make a report at once without waiting for further replies to the circulars of inquiry sent out to the members of the association. The recommendations of the committee are as follows:

(1) That the nomenclature now in use for fertilizers, soils, ash, etc., be retained.

Adopted.

(2) That the association vote upon the advisability of permitting the use of a dual system of nomenclature, when desirable, with a view to the ultimate adoption of the element system for reporting the analysis of fertilizers, soils, ash, etc.

After considerable discussion Mr. Davidson called attention to the fact that this motion merely called for a vote for the consideration of the subject and did not commit the association to the support of the dual system of nomenclature. The motion was put and the second recommendation carried.

Following the adoption of these motions Mr. Hopkins spoke at some length in regard to the difficulties inherent in the present system of nomenclature, and read a number of letters received favoring the element system. An abstract of Mr. Hopkins's remarks is as follows:

MR. HOPKINS. In presenting some of the arguments in favor of the element system of nomenclature for soils and fertilizers, I may first mention briefly the action already taken.

In 1904, at the St. Louis meeting of the Association of Official Agricultural Chemists, a committee was appointed on unification of terms used in reporting analyses; and at the Des Moines meeting a similar committee was appointed by the Association of American Agricultural Colleges and Experiment Stations. These two committees have cooperated in the work assigned and at the annual meetings in 1906 made substantially the same report to their respective associations.

Final action on the report was postponed for one year by the Association of Official Agricultural Chemists at the Washington meeting in 1906, in part because at that time no action had been taken by the broader Association of Agricultural Colleges and Experiment Stations, whose annual convention was held a week later at Baton Rouge, at which meeting, however, the report, which includes the following statements, was adopted with the conditions stated:

The subject-matter referred to your committee naturally divides itself into two classes, one of which includes soils, fertilizers, ash, and other materials whose analysis may be expressed in terms of chemical elements or in simple compounds; while the other class includes foodstuffs, condiments, and other materials whose analysis may best be expressed in terms of more complex compounds (or groups of compounds) which actually compose or are contained in the material.

SOILS, FERTILIZERS, ETC.

Providing concurrent action is taken by the Association of Official Agricultural Chemists and the American Chemical Society, your committee favors the adoption of the element system for reporting analytical results in the analysis of soils, ashes, and fertilizers, as rapidly as possible, and recommends that the association urge those responsible for fertilizer legislation to have the laws changed, if necessary, and as soon as practicable, to meet with these recommendations, if concurred in.

This would necessitate the adoption of the dual system of expressing results temporarily in some States, but it is hoped that when fertilizer laws are adopted to meet these requirements some definite time will be set, at the expiration of which only the element system will appear on the bags or tags. It is therefore suggested that no other terms than those of the element system be allowed after the year 1916.

Your committee also recommends, provided the foregoing is adopted, that this association adopt some definite form for stating the composition of fertilizers and fertilizer materials.

We recommend that the terms "available" and "inert" shall be used in harmony with the construction placed upon them by the Association of Official Agricultural Chemists.

The committee also recommends that in case of the adoption of the foregoing there be required to be printed on the bag or on the tag to be attached to the bag or to accompany fertilizers sold in bulk an explanatory statement naming the materials in which the plant food is carried.

This report, with the conditions stated as presented by the committee, was adopted by the Association of American Agricultural Colleges and Experiment Stations at the Baton Rouge meeting.

You will all agree that it is desirable, where possible and practicable, to report in analytical statements constituents which are actually present in the material analyzed and which are of direct interest to the party for whom the analysis is made.

In fertilizers, such as dried blood, sodium nitrate, ammonium sulphate, or calcium cyanamid, the only constituent which is actually present in each material and which interests the users of fertilizers is the element nitrogen, and in terms of nitrogen a comparison can be made of these materials which would not be easily possible if the analytical statements showed nitrogen (N) in dried blood, ammonia (NH_3) or ammonium (NH_4) in ammonium sulphate, nitrogen pentoxid (N_2O_5) or the acid radicle (NO_3) in sodium nitrate, and possibly some other radicle or compound for calcium cyanamid (CaCN_2). There exists at present confusion in the matter of reporting nitrogen, although the tendency is clearly toward the use of the element system in this case. Under the fertilizer laws of several States nitrogen from any source must be reported in terms of ammonia (NH_3), and this is the most common method of statement in the wholesale markets involving organic nitrogen, but to report ammonia in sodium nitrate, or nitrogen pentoxid in ammonium sulphate, would be extremely confusing to say the least. The only rational and scientific basis providing uniformity is to report nitrogen in all of these materials in terms of the element.

In the case of potassium the same argument holds with equal force. Potassium is already being reported in terms of the element to a considerable extent, and the practice is increasing and probably will increase as rapidly as was the case in changing from ammonia to the element nitrogen. (See, for example, Bulletin No. 22 and others, of the Bureau of Soils; Bulletin No. 60 of the New Mexico Agricultural Experiment Station; Bulletins 182, 183, and others, of the Ohio Agricultural Experiment Station, and the experiment station bulletins and State fertilizer laws of Kansas and Illinois.)

The element phosphorus is most commonly reported in terms of the pentoxid (P_2O_5), which with chemical inaccuracy is termed "phosphoric acid." In most of the publications from the Bureau of Soils,^a and in the publication referred to from the New Mexico experiment station, phosphorus is reported in terms of the acid radicle (PO_4), while in the experiment station bulletins of Ohio, Kansas, and Illinois, it is reported as the element (P), and this is already

^a The following note by the chief of the Bureau of Soils is submitted in this connection to make clear the position of that Bureau in regard to the use of the element system:

* * * There seems to be a serious misapprehension on the part of several members of the association as to the position of the Bureau of Soils regarding the statement of analytical results. The Bureau of Soils follows the recognized practice of the U. S. Geological Survey and the foremost laboratories of this country and abroad. In stating the analytical results of analyses of water solutions the "ion" system, or the system founded on the hypothesis of electrolytic dissociation in aqueous solution, is employed. In other statements of mineral analyses the conventional "oxid form" is employed. In some few cases the "ion" system appears to conform to the proposed "element" system, but the conformity is by no means real. The "element" system has never been employed by the Bureau of Soils, nor has it ever seemed necessary or desirable to express an official opinion concerning it. * * *

required by the fertilizer laws of two States. Phosphorus in fertilizers is also very commonly reported in terms of tricalcium phosphate, ordinarily known as "bone phosphate" or "lime phosphate" or "bone phosphate of lime." In many cases this is objectionable because the compound does not exist in the fertilizer, as in acid phosphate, dissolved bone, or basic slag.

In the matter of soil analysis almost insurmountable difficulties are met with in attempting to report the elements present in terms of compounds on a uniform comparable basis.

Berthelot and André^a report having found sulphur in the soil in the form of mineral sulphates, ethereal sulphates, metallic sulphids, and in organic nitrogen compounds of sulphur. They also report that more than one-half of the total sulphur of the soil is sometimes lost in ash determinations, and yet in most published reports of soil analysis sulphur is reported in the form of SO_3 whether contained in the soil in the form of sulphids or organic sulphur or as mineral sulphates.

In a recently issued text-book on soils, in four consecutive statements of soil analyses iron is reported as "ferric oxid," as "iron sesquioxid," as "iron protoxid," as "peroxid of iron," and as "oxid of iron." In volume 1 of the *Cyclopedia of American Agriculture*, published in 1907, manganese is reported as "manganese oxid (MnO)," as "manganese (Mn_2O_4)," as "manganese oxid (Mn_2O_3)," and as "manganic oxid (MnO)." In the same publication are soil analyses in which the sulphur is reported as "sulphuric acid," as "sulphuric anhydrid," as "sulphurous acid," and as "sulphuric trioxid."

There is but one simple basis for reporting an ordinary soil analysis and that is the basis of the chemical elements present. If necessary, we can report ferrous iron and ferric iron, which will give strictly comparable data, whereas the equivalent weights of ferrous oxid and ferric oxid are not directly comparable. Likewise we can report organic phosphorus and mineral phosphorus; sulphur in sulphids, in sulphates, and organic sulphur; also organic nitrogen, ammonia nitrogen, and nitrate nitrogen, and in all cases comparisons are direct and plain. To report the equivalent weights of NH_4 and NO_3 in ammonia salts and nitrate salts, because those radicals happen to be cations or anions, renders direct comparison impossible and serves no purpose of value to agriculture.

Think of reporting to the farmer that his soil is acid and in need of lime—perhaps 5 tons to the acre—and at the same time sending him an analysis showing that his soil contains 1 per cent of calcium oxid from which he finds by easy computation that his soil already contains 18 tons of lime per acre-foot. Then explain to him that the analytical statement is not correct, that his soil contains no calcium oxid, but that it contains sufficient calcium to make 1 per cent of calcium oxid if it were to be converted into calcium oxid, but into which it will not be converted, and that consequently his soil remains acid and does need lime.

Think of reporting an analysis of potassium chlorid as 63 per cent of potassium oxid and 48 per cent of chlorin (less oxygen misplaced, 11 per cent), and yet we have been doing just such unnecessary and unreasonable things in agricultural chemistry; meanwhile the farmers and farmers' institutes and the agricultural press continue to call upon science for the plainest, simplest statement of facts with the least possible use of unnecessary technical terms or expressions.

^a Ann. chim. phys., 1888, 15 [6]: 119.

In the chemistry of iron and steel when we deal with phosphorus we say phosphorus and we mean phosphorus, and in pharmacy and medicine when we say phosphoric acid we mean phosphoric acid, but in agricultural chemistry when we deal with phosphorus, organic or inorganic, we say and write phosphoric acid and we mean usually phosphorus pentoxid or sometimes the radicle of phosphoric acid (PO_4), but never true phosphoric acid unless we speak as the farmer's druggist, physician, or veterinarian, and then it should be understood that we mean phosphoric acid.

If we desire to report the oxygen in a soil, we can report the total amount by computation and by difference just as accurately as we do under the old system. As a matter of fact, few of the elements in soil analysis are weighed as oxids, most of them being determined either by volumetric processes or by the gravimetric determination of some salt, as, for example, barium sulphate, potassium platinic chlorid, magnesium pyrophosphate, etc.

If the interest in any constituent concerns the compound present, then, in addition to the statement of elements, there should be given the percentage or amount of such compound. Thus the calcium carbonate present in the soil may be reported as such, not because of any element of plant food which it contains, but because of its value as limestone. Indeed, this is already being done in cases where the importance of calcium carbonate, as such, is recognized.^a

The only valid objection to the adoption of the element system for reporting analyses of soils and fertilizers is that most analyses have been previously reported on the basis of oxids, and consequently there would be some additional burdens, temporarily, in making comparisons with earlier work until the more important existing data shall have been compiled and recalculated to the element basis. This objection, which will apply to a few people for a few years, is extremely slight compared with the advantages of the simpler system to all people for all time; and it is worth while to call to mind in this connection that our forefathers discarded "pounds, shillings, and pence" for the decimal system, with its dollars and cents, at a time when the change concerned not a small proportion but practically the entire population of the country; also, that nearly all other countries, except Great Britain, have subsequently adopted a decimal money system; and, not only that, but they have also adopted decimal systems of weights and measures, a change involving temporary inconvenience and expense for almost every business in the country.

International agreement in the adoption of the element system for soils and fertilizers is desirable; but, even though the element system should never be adopted in European countries, it is nevertheless highly desirable that it should be adopted in this new agricultural country and that American agriculture should have the benefit of using the simplest basis in the application of science to this fundamental industry, upon whose ultimate success practically all other great industries depend.

The advantages of the simple element system for soils, fertilizers, and plant-food elements in farm produce are so great that no State having once adopted the element nitrogen in place of ammonia has ever returned to the ammonia basis, and for the benefit of her own agriculture any single State can adopt with advantage and profit the more complete element system, even though other adjoining States may retain some other system.

The association adjourned until 2 o'clock.

^a *J. Agri. Sci.*, December, 1907, 2: 306.

FRIDAY—AFTERNOON SESSION.

The discussion of the adoption of a dual system of nomenclature looking to the adoption of the element system for reporting analyses of fertilizers, soils, etc., was resumed, and Mr. Hopkins offered the following motion:

That the suggestion of the committee looking toward the ultimate adoption of the element system be approved, but that no State should discontinue the use of the terms now in use until such discontinuation is also approved by this association and that meanwhile the subject should be brought before the International Congress of Applied Chemistry in an effort to secure international agreement;

That this association appoint a committee to bring the matter of reporting results according to the element system before the next International Congress of Applied Chemistry with power to add to their numbers at their discretion.

A lengthy discussion followed the seconding of this motion, during which the following opinions were expressed:

H. W. WILEY. I favor referring the matter to the International Congress of Applied Chemistry. It must be remembered that the elements are not present as such in the fertilizers, and the labels should show the definite composition of the package. Ethically this is always the requirement for a true label, but a statement might be made that the materials present equal so much phosphorus or nitrogen.

EDWARD GUEDEMAN. It seems to me that a distinction should be made between the reporting of fertilizers and soils. Commercial conditions and common practice should be considered, and I can not see that the farmer will be benefited or obtain any more information from the element system than from the present one. The Council of the American Chemical Society is to consider this question at the December meeting and I shall oppose the adoption of the element nomenclature.

B. W. KILGORE. It seems to me that before going to the International Congress we should decide whether or not as an association we indorse the element system, and the motion as submitted does not seem to simplify the matter sufficiently. I can not see that the association comes any nearer a solution of the problem by sending such a report to the International Congress.

B. L. HARTWELL. Would it not be well to adopt the element system provisionally upon its adoption abroad and to send a committee to represent the association, approving such action if it can be made international?

H. J. WHEELER. I am in favor of the association calling the matter up and of the adopting of the element system if it can be made uniform usage under the State fertilizer law and by international agreement. In other words, I am opposed to the adoption of the element system under present conditions, but would approve it if universally adopted.

C. G. HOPKINS. It is not expected that any State would discontinue the use of its present system until further action is taken.

G. S. FRAPS. I am not willing to make this conditional indorsement of the element system. I believe that the association should decide this matter for itself and not pass it on to some other body for decision. The States accustomed to the old system will not be willing to begin their educational work over again in order to introduce a system which, to say the least, does not offer any advantage over that in use. We have uniformity practically all over the world in this

matter of expressing analyses of soils and fertilizers, with the exception of the States of Illinois, Ohio, and Kansas. It would be easier for these few States to abandon their new methods than for the remainder of the world to change methods of nomenclature which have been in use ever since the analysis of soils and fertilizers was begun. I endeavor to stand on the side of progress, and have honestly tried to see in what the merit of the elementary system of nomenclature consists, but it appears to me that its simplicity is a matter of words rather than facts, and that the old system is entirely preferable.

J. T. WILLARD. Kansas has adopted the element system without waiting for further action by the association. It makes no difference to the chemist or to the fertilizer manufacturer in respect to calculations how the results are stated. The farmers will require instruction in any case and can learn one system as well as another. Those who have learned the old system would be inconvenienced temporarily in learning the new. In the States where fertilizers have just come into use it would appear to be simpler to use the same terms in instructing the farmers, through bulletins and at institutes, as are used in instructing their children who study chemistry in our high schools and colleges. My plea is for the use of the right name on the label even at some temporary inconvenience, and not to saddle future generations with the present antiquated system. Had our forefathers been willing to endure a little difficulty in the introduction of the metric system we and our children would not be burdened with our complex and confusing tables of weights and measures.

B. W. KILGORE. The students in our agricultural colleges must understand the terms as at present used, and if they are to make any use of the literature on the subject they must deal in these terms.

B. B. ROSS. In response to a circular letter with reference to this subject which was sent out several years ago I expressed an opinion favorable to the use of the element system in reporting the results of analysis of soils and ash, but so far as its employment in reporting fertilizer analyses is concerned, I think it would be very unwise to take such a step for some time to come, and I do not think we should go on record as an association in favor of abandoning the present method of reporting fertilizer results.

Alabama was the first State to adopt the use of the term "nitrogen" instead of "ammonia" (NH_3), and we still find it necessary to explain the relation between the two. The authorities in our State connected with the fertilizer control have had great difficulty in securing the enactment of fertilizer legislation which is practically uniform with that of four or five adjacent States, and hence they are opposed to any change in the present system of reporting fertilizer analyses. For these reasons I must vote in opposition to the adoption of the element system for reporting results of fertilizer analyses.

J. G. LIPMAN. Many laws become obsolete when they lose the moral support of the people for whom they are framed. In this, as in other matters, the line of least resistance is always followed. Rules laid down for the guidance of men of science may become obsolete for the same reason. Now, there is evidently a strong sentiment in favor of the element system among some of the members of the association, and pronounced opposition to it among others. In adopting a dual system we give the element system a provisional approval. It can be discontinued when found inexpedient.

Only those chemists engaged in official fertilizer control work having a right to vote according to article 2 of the constitution, a rising vote was taken and the motion carried by a vote of 23 to 14.

The committee on the President's address, 1906, offered its report, which was accepted by the association.

REPORT OF COMMITTEE ON RESOLUTIONS.

Your committee submits the following report and recommends the adoption of the resolutions presented:

(1) Within the past few weeks Dr. George C. Caldwell, of Cornell University, and Dr. W. O. Atwater, of Wesleyan University, have been removed by death. Both these men were pioneers in agricultural chemical teaching and investigation in America, and as such have exerted a deep, abiding influence upon the progress of agricultural chemistry not only by their personal work but widely through the many who as students have received inspiration from personal contact with these men. Doctor Caldwell was an active member of our association as long as his health permitted, serving in the presidency one term and several times acting as referee. While Doctor Atwater was not directly active in our work, he was always in full sympathy with it. Many of us feel a distinct sense of personal loss in their death.

Be it therefore resolved, That we, as an association, express our sorrow at the removal of Doctor Caldwell and Doctor Atwater from earthly activities, and also our profound appreciation of the fundamental work which they performed as pioneers in agricultural chemistry.

Be it further resolved, That the secretary be requested to prepare for insertion in our proceedings appropriate minutes regarding the work of Doctor Caldwell and Doctor Atwater. (This resolution was adopted by a rising vote.)

(2) Whereas it is the belief of this association that great good may be accomplished for the unification and improvement both of analytical methods and of definitions of standards for the foods and drugs that enter into international commerce, by an international conference of chemists and others connected with food and drug control and with the administration of laws relating thereto;

Therefore, be it resolved, That this association favors such an international conference and requests the proper officials of the United States Government to take the initiative therein. [Resolution adopted.]

(3) *Resolved*, That the changes suggested by President Street in his address be adopted as follows:

That one day of our convention be devoted entirely to the reading of papers and their discussion, and that for this purpose the convention be divided into three sections covering the same subjects as our present committees on recommendations, and that all papers to be read in these sectional meetings be referred to the proper committee, with full power to reject the same or assign places to them in the programme sent to all members prior to the convention. [Resolution adopted.]

Signed: L. L. VAN SLYKE,
CYRIL G. HOPKINS,
W. A. WITHERS,
Committee on Resolutions.

REPORT OF COMMITTEE ON AMENDMENTS TO THE CONSTITUTION.

(1) Your committee respectfully recommends that Article IV of the constitution shall be amended by the insertion in the second line, after the word "meeting," of the clause "from among the members of the association."

[This recommendation was adopted, and in accordance with the suggested change Article IV of the constitution reads as follows:

There shall be appointed by the executive committee at the regular annual meeting, *from among the members of the association*, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.]

(2) With respect to the suggested change in the tenure of office and organization of the committees on recommendations, your committee believes that, for the present, the matter could be better disposed of by the adoption of a resolution than by the insertion of a new article in the constitution.

It recommends for the attainment of the chief purposes of the proposed changes, the adoption of the following resolution with its preamble:

Whereas it is desirable to secure the greatest practicable uniformity in the form of expression and arrangement of the methods adopted by this association, and also to provide for the thorough consideration of the various changes in methods and of new methods proposed;

Resolved, That, beginning with 1908, the committee on recommendations be composed of nine members, elected (*appointed by the president*) as follows: Three to serve for six years, three for four years, and three for two years; and that as their respective terms expire, the vacancies caused thereby be filled by the election (*appointment*) of qualified successors for the full term of six years.

Resolved further, That this committee shall organize from its membership three subcommittees, corresponding to the present committees A, B, and C, to whom all recommendations of new methods or of changes in methods, provisional or official, shall be submitted not later than three weeks prior to the date of the meeting of the association at which it is desired that the recommendations in question shall be considered, and said committee on recommendations shall report to the association, with its approval, disapproval, or suggestion of amendment all recommendations submitted in due form for its consideration.

[The difficulty of electing such a committee by the association as a whole and insuring that the members be well chosen, and also the method of filling vacancies occurring during the year, were discussed at some length and a motion to amend this resolution to read "appointed by the president," instead of "elected," was passed. The original resolution as amended was then adopted.]

(3) With respect to the suggested expansion of the territorial extent of this association, your committee most cordially seconds the appreciation of good fellowship and of mutual scientific advantages gained by the attendance of our Canadian confrères in this and earlier years, and further is convinced that many advantages would result from the harmonizing of the official control methods and standards used by our North American neighbors and ourselves. The changes suggested are, however, far-reaching in their effects and may change the status of the association in ways not intended. It is believed that before action the subject should receive full consideration.

We therefore recommend that the following amendments to the constitution be brought to the notice of the association in the published announcement of the next annual meeting for the action of that meeting:

In Article I, first sentence, substitute for the words "the United States" the words "North America."

In Article II, first sentence, second and third lines, insert the word "provincial" after the word "State;" also in the third line of this sentence insert after the word "body" the phrase "in North America."

[This recommendation was adopted, and the proposed amendment is placed on record for action at the 1908 meeting.]

WM. FREAR, *Chairman*.
B. W. KILGORE.
J. G. LIPMAN.

Col. J. Rice Smith, then addressed the meeting and spoke most enthusiastically of the chemist's work in its relation to the fertilizer trade:

This is the body of men to whom we look for the information which it is so necessary and important for us to have in the proper conduct of our business, and you are of more value to us than the lawmakers in Congress. We are working along the same lines and for the same purposes—to develop and build up the industries of this country, and on our part especially the agricultural industry. Everything that we buy goes through the laboratory and everything that we sell does the same, that we may know what we are getting and what we are giving. Our chemist is at the top of the building—and that is where he belongs in relative importance. It is the laboratory first and last with us, and I do not flatter you, gentlemen, when I say this association is of more value to us than any other body of men. We have problems to meet, and only the chemist can solve them for us. In order that we may give every one the value of his money and have our material uniform and true to label, the chemist must furnish us with the facts and we can put the information to intelligent and practical use. The chemist is always showing us how to turn our waste material into wealth. Thirty or forty years ago cotton seed—then the burden and waste of our great cotton crop—were in the way or thrown away, while to-day, throughout the civilized world, millions of dollars' worth of meal, oil, and other products are made from this source and utilized for feeding, fertilizing, and domestic purposes. It was only through the science of chemistry and the work of the chemist that this result could have been accomplished. The possibilities lying dormant in this country, if we develop them, will enable us to clothe and feed the world, and it is to chemistry that we must look for the information that will enable us to do it. Any improvement that you can suggest regarding our industry we will be glad to act upon. I thank you for your courteous attention.

REPORT ON PHOSPHORIC ACID.

By B. W. KILGORE, *Referee*.

The amount of American data available for determining the fertilizing value of Thomas or basic slag in comparison with other materials furnishing phosphoric acid is not large, but the territory over which the material is used and the amount employed for fertilizing purposes are annually increasing. It is therefore desirable to adopt a tentative or provisional method for its analysis and fix a basis for stating its commercial value. Until further data are obtained in connection with experiments which are now in progress or which may be instituted in the future the following recommendations are made:

RECOMMENDATIONS.

It is recommended—

(1) That Thomas or basic slag be prepared for analysis as other fertilizer or fertilizing materials are under the methods of this association.

(2) That the solution for analysis be made according to a-7, Bulletin 107, page 2, or in strong hydrochloric acid alone. In the latter case, after the portion for analysis is measured out, add nitric acid and heat for a few minutes. Then determine total phosphoric acid according to the official methods.

(3) That the fineness of the material be determined according to the plan followed with bone meal and the commercial value estimated on the basis of total phosphoric acid and fineness of product.

SUBREPORT ON PHOSPHORIC ACID.

By J. M. McCANDLESS, *Referee*.

On June 3, 1907, the following letter, together with one sample of Tennessee phosphate rock and two samples of acid phosphate, was sent to about twenty analysts, who had expressed a willingness to cooperate in the work:

DEAR SIR: I have sent you by express a box of samples for the cooperative work of the association on phosphoric acid and should be pleased to receive results on this work at your earliest convenience. To those who are interested in the question of the effect of sulphuric acid on iron pyrites in phosphate rock I would recommend that they add to a weighed portion of the rock 10 per cent of its weight of high-grade iron pyrites, then treat with sulphuric acid as described in (1) and determine whether any additional iron has passed into solution from the pyrites. This idea occurred to me too late to inclose a sample of the pyrites with the other samples.

METHODS TO BE USED IN THE DETERMINATION OF IRON AND ALUMINUM IN PHOSPHATE ROCK.

It is recommended that before beginning the work on a sample of phosphate rock a solution be made up imitating a phosphate rock solution and containing known quantities of iron and aluminum.

Make the solution about as follows:

Weigh 18.25 grams microcosmic salt; 0.400 gram iron wire; 8 grams recrystallized potash alum; 12.5 grams C. P. calcium carbonate; 0.300 gram calcium fluorid. Transfer to a liter flask, add dilute hydrochloric acid till carbonate is all in solution, and then more hydrochloric acid to dissolve the iron. Heat till solution is complete, cool, and make up to mark with distilled water.

It is earnestly recommended that the following methods be tried for your own satisfaction on this solution, in which you will know the percentage of iron and aluminum, before beginning work on the sample of phosphate rock.

I send only one sample of Tennessee phosphate rock, desiring to make the work as light as possible, and thinking it better to apply all the methods to one sample rather than to send a number of samples, which would involve much labor and probably result in less valuable work being done.

Inasmuch as in the manufacture of acid phosphate the rock is nearly always treated with about its own weight of 50° B. sulphuric acid, and as, where the rock contains clay, more Al_2O_3 is extracted by this treatment than is usually dissolved by hydrochloric acid in the methods of analysis prevailing, I have thought it wise to imitate the process of manufacture and to treat the rock in all the following methods with 50° acid before bringing it into solution with the hydrochloric acid.

Methods for alumina.

(1) *Thiosulphate method, modified*.—Treat 2.5 grams of the sample of phosphate rock (previously treated with a magnet to remove small particles of iron derived from the pulverizing implements) with an equal weight of sulphuric acid (sp. gr. 1.53) and heat on water bath for thirty minutes. Boil with 50 cc of 1:1 hydrochloric acid for thirty minutes and make up to 250 cc. Filter off 100 cc, equivalent to 1 gram, add about 100 cc of water, and bring to a boil. Remove from flame and add a slight excess of ammonia. Clear with hydrochloric acid, added drop by drop with constant stirring, then add 2 cc of concentrated hydrochloric acid in excess. Add 10 grams of sodium hyposulphite and 15 cc of acetic acid (sp. gr. 1.04) and boil for fifteen minutes. Filter on a rapid ashless filter paper and wash with hot 5 per cent ammonium nitrate solution. Ignite at a low temperature till paper is charred and then increase the heat gradually till carbon and sulphur are oxidized. Finally blast for one minute and weigh as phosphate of aluminum, containing 41.85 per cent of Al_2O_3 .

(2) *Gladding method*.—Treat 2.5 grams of the sample as above with an equal weight of sulphuric acid (sp. gr. 1.53). Heat with 50 cc dilute hydrochloric acid (1:1) and keep just below the boiling point for thirty minutes. Make up to 250 cc and filter off 100 cc (1 gram). Add a few drops of nitric acid to oxidize the iron and boil for a few minutes. Run this solution into 20 cc of a solution of C. P. caustic potash, made by dissolving 500 grams of caustic potash, free

from alumina, in distilled water and diluting to 1 liter. Digest in water bath at 70° for one hour, stirring occasionally. Let the precipitate settle, and filter on a large filter, first decanting the supernatant liquor on the paper, and finally transferring the precipitate to the filter. Wash the precipitate two or three times with hot water. To the filtrate add 1 gram of ammonium phosphate, acidify with hydrochloric acid, add ammonia till a permanent precipitate is formed, and then dilute hydrochloric acid drop by drop until it is just dissolved. Add a mixture of 15 cc of neutral ammonium acetate and 5 cc of acetic acid (30 per cent) and digest for half hour at 70°. Filter, washing five or six times with hot 10 per cent ammonium acetate solution, stirring up the precipitate each time with the jet from the wash bottle. Ignite with a low flame till the paper is charred. Increase the heat, and when the paper is entirely consumed blast for one minute. Weigh and calculate the percentage of Al_2O_3 as in (1).

(3) *Glaser method*.—Treat as in (1) up to the point when 100 cc, or 1 gram, has been filtered off. Add 25 cc of concentrated sulphuric acid, shake, and allow to stand a few minutes. Add about 100 cc of alcohol and cool. Make up to 250 cc with alcohol and allow to stand thirty minutes. Filter off 100 cc, equivalent to 0.4 gram. Evaporate to expel alcohol, using a rather large, hard glass beaker. Transfer to a smaller beaker and add a few drops of nitric acid to oxidize the last traces of alcohol and boil. Remove from the flame and cautiously add ammonia to slight alkalinity. Boil to neutrality; allow to settle and cool. Filter and wash with hot ammonium nitrate solution (10 per cent). Burn and weigh. Deduct from the weight the iron phosphate found volumetrically, to obtain the percentage of $AlPO_4$, or divide the weight found by 2 to obtain the per cent of the combined oxids of the iron and aluminum.

Oxid of iron.

Use any volumetric method you prefer for the determination of the iron.

The following method has been used by us and found to be very satisfactory:

Take 100 cc of the filtered solution used in the determination of the alumina under (1), transfer to a 500 cc flask, add a few crystals of potassium chlorate, and boil till chlorin has been removed. Add 3 grams of granulated zinc and 5 cc of hydrochloric acid. Place a funnel in the mouth of the flask and heat slightly till the iron has all been reduced and the zinc nearly all dissolved, then add 10 cc of sulphuric acid diluted with 20 cc of water, and boil till all the zinc has gone in solution. Make the solution up to about 500 cc with cold water and pour into 500 cc of cold water in a large white dish. Rinse the flask and add the washings to the dish. Titrate with standard potassium permanganate, making a correction for the iron in the zinc.

It is desired that you determine the percentage of insoluble phosphoric acid in the two samples of acid phosphate sent, using, first, the ammonium citrate as usually made in your laboratory; secondly, a solution of ammonium citrate made according to the following method:

Weigh out a suitable quantity of citric acid and calculate the approximate amount of ammonia that should be added to it to neutralize it. It has been our practice to weigh 7.5 pounds of citric acid, then pour on about 2 liters of water and add 3,100 cc of ammonia (0.90 sp. gr.) to it. Stir till all the acid has been dissolved and allow to cool. The solution gets very hot, and there is a loss of ammonia, leaving the solution acid. Make up to a definite volume, about 10 liters. Measure carefully 25 cc of this solution into a 250 cc flask, make up to the mark with distilled water, measure out 25 cc of this solution (corresponding to 2.5 cc of the original solution), and transfer to a distilling flask. Add an excess of standard potassium or sodium hydroxid and distil the ammonia into standard acid. Titrate and calculate the amount of ammonia, and when cool titrate the excess of caustic alkali in the residue in the distilling flask, using phenolphthalein as indicator, and calculate the amount of citric acid. Knowing the amount of ammonia and citric acid in the measured volume, calculate the quantity of ammonia necessary to neutralize the excess citric acid in the main body of the solution. Add this quantity from a solution of ammonia of known strength, shake and bring to a specific gravity of 1.09. This solution made neutral by analysis reacts alkaline both to litmus and to corallin. It also shows alkaline by the calcium chlorid test, but it will be found on trial that though most of the citric acid is removed from the ammonium citrate by alcoholic calcium chlorid, that a notable quantity still remains in solution, and that therefore this test is not so reliable as has been thought,

Unfortunately, pressure of other duties must have prevented the analysts from cooperating, as only the following results on iron and alumina had been received:

TABLE I.—*Determination of alumina and ferric oxid.*

(a) SYNTHETIC MIXTURE.

Analyst.	Sample.	Determination.	Method.	
			Gladding.	Thiosulphate.
Stillwell and Gladding (New York City).	{ Synthetic mixture.	{ Alumina (Al_2O_3)..	<i>Per cent.</i>	<i>Per cent.</i>
			12.42	12.23
			12.44	12.48
			3.03	3.05
McCandless and Burton (Georgia State Laboratory).	{ 3 per cent Fe_2O_3 ; 5 per cent Al_2O_3 .	Ferric oxid (Fe_2O_3)..		
		Alumina (Al_2O_3)..	5.10	5.07

(b) TENNESSEE ROCK.

Analyst.	*Alumina (Al_2O_3).			Ferric oxid (Fe_2O_3).
	Gladding method.	Thiosulphate method.	Glaser method.	
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
McCandless and Burton (Georgia State Laboratory).	5.40	5.35	5.81	3.94
	5.32	5.40		
	6.44	4.73		
Stillwell and Gladding (New York City).....	6.74	4.95		
	5.27	5.38	5.33	
J. H. Mitchell (Clemson College, S. C.).....	5.27	5.48	5.28	4.54
	5.33	5.53	5.25	
F. B. Carpenter (Virginia-Carolina Chemical Company, Richmond, Va.).....		a 5.34		b 4.48

^a Average of seven determinations.

^b Obtained by calculating all the percentages of Al_2O_3 .

It is unfortunate that a greater number of chemists did not engage in the work, as the results of three of the chemists compare favorably on the percentage of alumina by the first two methods, but are offset by the wide difference in the results of the author of the Gladding method.

Subsequent experience by the referee has, however, shown that the thio-sulphate method will, with certain rocks, give results that are far too low, and that it can not, therefore, be recommended in its present form. The simplicity and rapidity of the method, however, make it very desirable that the next referee continue work on it and endeavor to ascertain the cause of the low results obtained with certain phosphate rocks. At first the referee believed this to be due to the presence of fluorin, but the excellent results obtained in synthetic mixtures containing varying quantities of fluorids, discredit this view.

The referee added to three samples of finely ground rock 10 per cent of their weight of high-grade pyrites and then treated the mixture as outlined in Method (1). Careful determinations of iron in the resulting solutions showed not more than 0.1 per cent additional of ferric oxid to have been brought into solution, confirming the results of experiments made some years since by the referee and published in the American Fertilizer. Unfortunately, no other chemist to whom samples were sent reported any experiment along this line.

Two samples of acid phosphate, Nos. 2 and 3 in the following table, were sent out with the request that the insoluble phosphoric acid be determined, first, with ammonium citrate as usually made in the laboratory, and, secondly, by

the solution made as prescribed by the referee. Unfortunately, only two other chemists besides the referee and his associate have reported results on these two samples.

TABLE II.—*Determination of insoluble phosphoric acid.*

Analyst.	Ammonium citrate.		
	Slightly acid to litmus.	As ordinarily made with corallin.	Made neutral by analysis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
	SAMPLE NO. 2.		
H. L. Davidson, Virginia Department of Agriculture.....	6.42	6.88	6.98
Robert H. Kerr, Lazaretto Guano Works.....		6.16	6.20
McCandless and Burton, Georgia Department of Agriculture.....	4.12	5.90	6.23
F. B. Carpenter, ^a Virginia-Carolina Chemical Company, Richmond, Va.....	{	7.00	7.12
		7.05	7.10
		7.05	6.99
	SAMPLE NO. 3.		
H. L. Davidson.....		4.08	4.11
Robt. H. Kerr.....		3.72	3.70
McCandless and Burton.....	4.15	4.23	4.22
F. B. Carpenter, ^a Virginia-Carolina Chemical Company, Richmond, Va.....	{	4.06	4.05
		3.98	4.00
		4.10	4.07

^a Analysts, Henry and White.

It has been the experience of the referee that with the average acid phosphate slight differences in the neutrality of the ammonium citrate solution cause little or no variation in the percentage of insoluble phosphoric acid, but that occasionally samples of acid phosphate are found in which slight variations in neutrality cause very wide variations in the percentage of insoluble phosphoric acid. Sample No. 2 was of this character, and the referee therefore regrets that a greater number of chemists did not cooperate in the work.

A paper was submitted by Mr. Carpenter giving some figures obtained by procedures varying from those outlined by the referee, and the results of these studies are submitted as a supplementary report as follows:

SUPPLEMENTARY REPORT BY F. B. CARPENTER.

When sulphuric acid is used in the preliminary digestion according to your instructions, the results seem to be more uniform and satisfactory than when solution is made simply with hydrochloric acid. We tried making solutions with both concentrated and dilute hydrochloric acid, but it did not seem that it made any material difference in the results. Boiling one-half hour with concentrated hydrochloric acid without previous treatment with sulphuric acid does not seem to be sufficient time to decompose all of the iron and alumina phosphates in the sample under consideration.

The modified acetate method as used to obtain the results in the table is as follows:

Weigh 2 grams of sample in a 200 cc flask, add 30 cc of strong hydrochloric acid c. p., boil half an hour. Dilute, cool, make up to mark, mix, pour through dry filter. Measure 50 cc into an 8-ounce beaker. Oxidize by boiling with a little nitric acid, remove the beaker from the flame, neutralize with ammonia, and then just dissolve the precipitate in diluted hydrochloric acid, adding drop by drop, taking care to add only sufficient acid to dissolve precipitate.

Add 30 cc saturated solution ammonium chlorid, add water, filling the beaker to within about three-fourths of an inch from the top, place in water bath and heat to 70°. Remove from bath, add 5 or 6 drops of methyl orange solution, and then add ammonium acetate solution which has been heated to 70° until color changes from red to yellow. Add an excess. Filter on folded filter, and wash two or three times with hot 5 per cent solution of ammonium nitrate; dissolve this precipitate back into the same beaker, using diluted hydrochloric acid and hot water. Add 1 gram of ammonium phosphate to the solution and precipitate with ammonium acetate, proceeding as before. Filter and wash the last precipitate with 5 per cent solution of ammonium nitrate five times and once with hot water.

TABLE III.—*Determination of alumina by the thiosulphate method and modifications.*

[The totals are averages.]

Method described by referee, thiosulphate method No. 1.	Method No. 1 with addition of 3 per cent calcium fluorid to flask after weighing sample.	Modified acetate method, solution made as in method No. 1.	
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
5.45	5.08	5.21	
5.30	5.07	5.18	
5.25	5.20	5.15	
5.51	5.08	5.10	
5.30	5.17	5.16	
5.25	5.00		
5.34	5.10		
5.34	5.10	5.16	

Solution made by boiling 2 grams of rock one-half hour with concentrated hydrochloric acid without previous treatment with sulphuric acid.		Solution made by boiling 2 grams of rock one hour with 50 cc of concentrated hydrochloric acid.	
Thiosulphate method.	Modified acetate method.	Thiosulphate method.	Modified acetate method.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
4.91	4.47	5.00	4.90
4.85	4.45	5.15	4.77
4.73			
4.83			
4.83	4.46	5.08	4.84

We have not done sufficient work by the modified acetate method to vouch for its accuracy, but we believe that with some slight modifications it will be an ideal method. The advantage this has over the regular laboratory method, which we have previously used, is the elimination of any considerable amount of lime from the precipitate. In method No. 1, unless great care is taken in the manipulation, there will be some lime left in the precipitate and the results will be correspondingly high. In the hands of different chemists, especially those with little experience, our method has not always given good results, but after having had considerable experience with the method the analysts usually get excellent results.

PRELIMINARY STUDIES ON THE ANALYSIS OF BASIC SLAG FOR AVAILABLE PHOSPHORIC ACID.

By H. D. HASKINS.

The slag used for the following tests analyzed 19.02 per cent, 18.94 per cent, and 19.02 per cent total phosphoric acid, or an average of 18.99 per cent.

In view of the fact that it is generally conceded that the beneficial effect of Thomas slag phosphate on growing vegetation may be due, in part at least, to

the free lime present in the slag, it occurred to the writer that the fairest way of treating the slag for the determination of the available phosphoric acid was first to neutralize the free lime and finally treat the resulting product with neutral citrate of ammonia in the usual manner. This was accomplished by the two following methods:

Method A.—Two grams of the Thomas phosphate powder were weighed into an Erlenmeyer flask of 150–200 cc capacity, 10 cc of distilled water and a few drops of phenolphthalein solution as an indicator were added. The flask was then placed on a boiling water bath and when the solution became thoroughly heated, dilute citric acid was run into the flask until the pink color disappeared. After the mass had been brought to dryness, 100 cc of neutral citrate of ammonia were added and the digestion was carried on at 65° C. in the usual way as for the determination of citrate-soluble phosphoric acid. This method showed 6.61 per cent and 6.29 per cent of available phosphoric acid.

Method B.—Two grams of the Thomas phosphate powder were transferred to a small beaker, 10 cc of distilled water and a few drops of phenolphthalein solution were added and the beaker was placed on a boiling water bath. When hot, an excess of dilute citric acid solution was added and the mass was digested for fifteen minutes on the hot water bath; the solution was then filtered into a 200 cc flask and washed with hot water until it was up to the 200 cc mark. The residue was treated with neutral citrate of ammonia in the usual way and showed 6.83, 6.37, and 6.60 per cent of available phosphoric acid. The citric acid solutions that had been filtered off showed about 0.06 per cent of phosphoric acid.

Another set of samples was run according to method B, only a larger excess of citric acid was used in the digestion. The citric acid solutions upon being analyzed showed 1.40, 1.66, and 1.48 per cent of phosphoric acid. The residues showed 6.13, 5.71, and 5.40 per cent of citrate-soluble or reverted phosphoric acid, making the available phosphoric acid in this case 7.53, 7.37, and 6.93 per cent. It was noticed when the residue from the citric acid solution was being washed with hot water, that after a few washings the water, as it left the residue, was of a pinkish color.

The sample of slag on which the above tests were made showed 47.83 and 47.72 per cent or an average of 47.77 per cent of calcium oxid of which 7.38, 7.40, and 7.43 per cent were actually found present as free calcium oxid.

No attempt was made to determine the available phosphoric acid in the above slag by the method in use at the California experiment station or by the Wagner method. It appears to the writer that of the two methods, Method B offers the most encouragement for development. Although not giving satisfactory concordant results, as above outlined, it would seem that if a given quantity of solution of citric acid of known strength were used in each case, satisfactory comparative results would follow. It is earnestly hoped that the association will investigate the matter of the determination of the available phosphoric acid in basic slag and adopt, at least provisionally, some method for the analysis of this product, the use of which is becoming general in certain sections of our country.

REPORT OF COMMITTEE B ON RECOMMENDATIONS OF REFEREES.

B. B. Ross, *Chairman.*

(Dairy products, foods and feeding stuffs, sugar, tannin, and medicinal plants and drugs.)

(1) SUGAR.

It is recommended—

(1) That in the determination of reducing sugars the use of lead subacetate as a clarifying agent be discontinued and that only those agents, such as normal lead acetate, be employed which do not precipitate reducing sugars.

Adopted.

(2) That the use of potassium oxalate for precipitating the excess of lead from sugar solutions before polarizing or determining reducing sugars be adopted provisionally.

Adopted.

(3) That the zinc oxid method for the determination of ash in molasses be dropped from the official methods. (Bul. 107, p. 68, Method II.)

Adopted.

(4) That the method and tables of Munson and Walker for the unification of reducing sugar methods be adopted provisionally by the association. (J. Amer. Chem. Soc., 1906, 28: 663; 1907, 29: 541.)

Adopted.

(5) That Low's volumetric method for the estimation of reduced copper (Bul. 99, p. 19) be made a provisional method of the association.

Adopted.

(6) That the question of the influence of normal lead acetate, of wet and dry lead subacetate, and of hydrosulphite upon the polarization of sugars be further investigated.

Adopted.

(7) That the analysis of commercial dextrans be further investigated by next year's referee on special analytical methods.

Adopted.

(2) FOODS AND FEEDING STUFFS.

It is recommended—

(1) That the method for methyl pentosans be further studied next year, especial attention being given to obtaining as much cooperative work as possible.

Adopted.

(2) That the table for calculating methyl-furfural-phloroglucid to fukose, fukosans, rhamnose, rhamnosans, and methyl-pentosans (average of fukosan and rhamnosan), given in an article by Mayer and Tollens (J. Landw., 1907, 55 (3): 269), be used in calculating the results of analysis; the average figure for methyl pentosans being taken when it is not known whether the mother product is fukosan or rhamnosan.

Adopted provisionally.

(3) That the last sentence under "Crude Fat or Ether Extract," "(b) (1) Direct Method," Bulletin 107, Methods of Analysis, page 39, which now reads as follows: "Dry the extract at the temperature of boiling water until it ceases to lose weight," be changed to read: "Dry the extract at the temperature of boiling water for one-half hour, remove from the oven to a desiccator, cool and weigh; continue this alternate drying and weighing at half-hour intervals until a minimum weight of fat is obtained. For most feeds a period of from one to one and one-half hours is required to obtain a minimum weight."

Adopted. [This is an elaboration of an official method and was adopted as a recommendation of the chairman of the committee on revision of methods, ordinarily two years being necessary for such action.]

(3) DAIRY PRODUCTS.

It is recommended—

(1) That the study of methods of analysis of condensed milk be continued and that special attention be given to the following methods for determining the fat and sugar content of condensed milk:

(a) Double extraction method for the determination of fat as described in the proceedings of the association for 1906.^a

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

(b) Roesse-Gottlieb method for the determination of fat as described in the proceedings of the association for 1906.^a

(c) Walker method for the determination of sugar. (J. Amer. Chem. Soc., 1907, 29: 541.)

(d) Allihn method for the gravimetric determination of sugar.

(e) Polariscope method in comparison with the gravimetric method for the determination of sugar.

Adopted.

(4) TANNIN.

It is recommended—

(1) That work be continued with a view to finding a cheap strong filter paper as a substitute for S and S 590.

(2) That the method of filtering soluble solids suggested by Reed be given further trial. (J. Amer. Leather Chemists Assn., 2: 420.)

(3) That the referee take up the study of methods for the analysis of leather, as such methods are greatly needed.

Recommendations adopted.

(5) MEDICINAL PLANTS AND DRUGS.

It is recommended—

(1) That the present provisional method for assaying opium be made official. (Bul. 107, p. 201.)

Referred to referee for recommendation as to final action in 1908.

(2) That the methods included in the referee's report be made provisional. (See p. 81.)

Adopted.

(3) That the work be continued and that the associate referees be appointed on the following subjects:

(a) Analytical methods for crude products.

(b) Methods for detecting and estimating alkaloidal matter in mixtures.

(c) Macroscopical and microscopical methods.

(d) Microchemical methods.

(e) Methods for headache mixtures.

(f) Methods for determining alcohol in the presence of other volatile bodies.

(g) Methods for testing volatile oils.

After some discussion this motion was put to a vote and was lost, it being deemed inadvisable to create associate referees for such divisions of the subject at this time, and the referee was instructed to conduct the investigations specified informally, assigning the several lines of work to such chemists as volunteered for cooperative work.

A motion was made to continue the committee for the testing of chemical reagents another year, and this motion was carried.

Mr. Kebler called attention to the fact that it was often desirable to use the text of the United States Pharmacopœia in connection with the association work on drugs, and this could not be done without the consent of the trustees of the Pharmacopœia. It was moved, therefore, that the secretary of the association be authorized to correspond with the proper authorities and secure this privilege. The motion was carried.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, p. 105.

REPORT ON POTASH.

By A. L. KNISELY, *Referee*.

At the last meeting of the association it was recommended that the study of the volumetric method for use in both soil and fertilizer analyses be continued. It was also recommended that the referee direct his attention to a study of what really constitutes available potash in soils, fertilizers, and ground mineral products, to the end that available potash might be defined.

The referee, assisted by Mr. C. E. Bradley, spent all of the time possible upon a study of the volumetric method, leaving the subject of available potash for consideration during the coming year. Considerable time was spent in preliminary experimental work upon details of the method before a satisfactory scheme was evolved as an outline for cooperative work.

In applying the method of Donk for estimating potash volumetrically, approximately correct results can be readily obtained. At times, however, discrepancies were noted in comparing volumetric values with those obtained by gravimetric methods. One of the vital points in the process is the use of a wash liquid which will remove all excess acid from the precipitated potassium phosphomolybdate and at the same time dissolve only inappreciable quantities of this yellow precipitate. Accordingly a test was made to determine the solubility of the potash precipitate in both nitric acid and sodium nitrate under different conditions in respect to strength, time of contact, etc. For this purpose a quantity of pure potassium phosphomolybdate was prepared by adding phosphomolybdic acid to excess of potassium sulphate in solution and washing the precipitate with a weak solution of sodium nitrate. To determine the action of nitric acid upon this precipitate, 0.1 gram of potassium phosphomolybdate was placed in contact with 50 cc of the nitric acid of varying strength, well stirred and filtered. The filtrate was evaporated to dryness on a water bath and the residue titrated with potassium hydroxid.

TABLE I.—*Determination of the solubility of the potash precipitate in nitric acid.*

No.	Conditions.	Strength of nitric acid.	Amount of potassium hydroxid required to neutralize.
			cc.
1	Heated to 50° C. and cooled 1 hour.....	5.5:1,000	1.00
2	One hour in cold.....	5.5:1,000	.10
3	Five minutes in cold with constant stirring.....	40 :1,000	.12
4	Ten minutes in cold with stirring.....	80 :1,000	.20

Heating to 50° C. is thus seen to exert marked solvent action, even with very dilute solutions of nitric acid. The cold acid of much greater strength exerts less solvent action, when time of contact is limited.

The action of the nitric acid wash under different conditions was also tested on an official acid (sp. gr. 1.115) extract of soil. Ten cubic centimeters of solution, corresponding to 1 gram soil, were evaporated with phosphomolybdic acid and the residue subjected to the following methods of washing:

TABLE II.—*Determination of the solubility of the precipitate of an official acid extract of soil in nitric acid.*

Sample.	Conditions.	Strength of nitric acid.	Potassium monoxid.
			<i>Per cent.</i>
1.....	Stirred in cold 5 minutes.....	5.5:1,000	0.41
2.....	Heated to 50° C. and cooled 30 minutes.....	5.5:1,000	.35
3.....	Heated 50° C. for 30 minutes and filtered after 5 hours.....	5.5:1,000	.23
		5.5:1,000	.24

The solubility of potassium phosphomolybdate in sodium nitrate solutions under different conditions was also investigated. A weighed quantity of the potassium phosphomolybdate was transferred to a gooch and 250 cc of sodium nitrate wash were run through as in analysis. The precipitate remaining in the gooch was then titrated with potassium hydroxid solution, 1 cc of the alkali used being equivalent to 0.0298 gram potassium phosphomolybdate.

TABLE III.—*Solubility of potassium phosphomolybdate in sodium nitrate.*

Sample.	Strength of sodium nitrate.	Phosphomolybdate.	
		Used.	Found.
		<i>Grams.</i>	<i>Grams.</i>
1.....	5:1,000.....	0.1461	0.1474
2.....	20:1,000.....	.0915	.0920

A negative result for solubility was thus obtained, probably due to a small error in the factor previously obtained. It is evident that a sodium nitrate wash of 2 per cent strength exerts very little solvent action on the precipitate of potassium phosphomolybdate under the conditions of the experiment. On long contact, however, a marked solvent action is evidenced by the yellow color which a solution develops when in contact with the precipitate. A test of a saturated solution gave results close to those noted by Fraps (1905).

It was also noted that pure water decomposes the precipitate, as stated by Donk, with the separation of a whitish acid substance, supposedly molybdic acid. In our hands a solution of sodium nitrate of the strength adopted (5:1,000) gave also a slight turbidity when left in contact with the precipitate for a short time. A solution of sodium nitrate (10:1,000) remains clear, however, under these conditions.

The preparation of the phosphomolybdic solution was also investigated, as it was noted that some solutions left no trace of insoluble residue when evaporated and taken up with nitric acid wash, while others left molybdic acid residues on evaporating, which required from 0.2 cc to 3 cc of standard potassium hydroxid solution to neutralize and which had to be allowed for as a "blank." Crystals of phosphomolybdic acid as furnished by Kahlbaum left no residue when the water solution was evaporated on a water bath and the dry residue again taken up with water. The nitric acid solution of the crystals, as recommended by Donk, when fresh left no residue under these conditions, but when allowed to stand some weeks left a residue from the nitric wash, which gradually increased in amount as the solution became older. The addition of a small amount of calcium and magnesium nitrate in the presence of a large excess of nitric acid seemed to prevent this, probably due to the formation of

soluble molybdates of calcium and magnesium with the separated molybdic acid. The presence of hydrochloric acid also prevented the formation of an insoluble residue, the hydrochloric acid combining with the molybdenum trioxid and forming a compound soluble in water or dilute nitric acid.

Phosphomolybdic acid solutions were prepared as follows:

- I. { 100 grams phosphomolybdic acid.
250 cc nitric acid (1.40 sp. gr.).
750 cc water.
- II. Duplicate of I (kept in dark).
- III. { 100 grams phosphomolybdic acid.
250 cc nitric acid (1.40 sp. gr.).
750 cc water.
10 grams calcium nitrate.
10 grams magnesium nitrate.
- IV. { 100 grams phosphomolybdic acid.
200 cc nitric acid (1.40 sp. gr.).
100 cc hydrochloric acid (1.20 sp. gr.).
700 cc water.
- V. { 100 grams phosphomolybdic acid.
40 cc nitric acid (1.40 sp. gr.).
960 cc water.
10 grams calcium nitrate.
10 grams magnesium nitrate.

These solutions were kept on the laboratory desk in rubber-stoppered Erlenmeyer flasks, with the exception of No. II, which was kept in the dark. From time to time 10 cc of each of these solutions were evaporated in beakers on the water bath to complete dryness—that is, until the odor of acid had completely disappeared. An examination of the dry residue taken up in the cold with nitric acid wash (50:1,000) resulted as follows:

TABLE IV.—*Solubility of residue from 10 cc phosphomolybdic acid solution in nitric acid (50:1,000).*

Solution.	July 24.	August 6.	August 20.	September 26.	October 3.	October 4. [Standard potash neutral- izing residue.]
I	Clear....	Trace precipitate	Trace precipitate	Small precipitate	Small precipitate.	cc. 0.10
II	Clear....	Trace precipitate	Small precipitate	Small precipitate	Small precipitate.	.10
III	Clear....	Clear.....	Clear.....	Clear.....	Clear.....	.00
IV	Clear....	Clear.....	Clear.....	Clear.....	Clear.....	.00
V	Clear....	Trace precipitate	Considerable white precipitate.	Heavy white precipitate.	Heavy white precipitate.	.70

These results show that much depends upon the age of the phosphomolybdic acid solution and upon the method of preparation. Fresh solutions are always to be preferred.

The accuracy of the method proposed for this year was checked by testing chemically pure potassium sulphate, with the result that in a sample containing theoretically 0.1284 gram of potassium monoxid 0.1279 gram was found, and 0.0398 gram was found as compared with 0.0399 gram present according to theory.

The evaporated residue from chemically pure potassium sulphate and phosphomolybdic acid is a fine amorphous powder which gave difficulty in filtering. The addition of a small amount of calcium or magnesium nitrate assisted in the formation of a crystalline residue which facilitated filtering greatly.

The presence of a soluble silica in soil extract did not interfere with the process, except that when the amount was large, filtration became troublesome from the clogging of the filter.

The use of the centrifuge for freeing the yellow precipitate of potassium phosphomolybdate from acid was tried, with good results. The use of a potassium hydroxid solution (1 cc = 1 mg K_2O) is probably preferable, but to avoid the labor of preparing two solutions the potassium hydroxid used in volumetric phosphoric acid work was utilized. As a result of all this preliminary work directions were formulated which gave good results in the station laboratory. The samples and directions were sent out as follows:

ASSOCIATION POTASH WORK, 1907.

Sample No. 1.—A sample of soil from the wheat-growing belt of eastern Oregon. This soil contains approximately from 0.25 to 0.5 per cent of acid-soluble potash.

Sample No. 2.—A mixed fertilizer containing considerable quantities of nitrogen, phosphoric acid, and potash (approximately 9 to 10 per cent of potassium monoxid).

WORK ON SOIL.

Mix thoroughly before sampling.

(1) Determine potash (K_2O) in soil according to the official method using hydrochloric acid, 1.115 sp. gr. (U. S. Dept. Agr., Bureau of Chemistry, Bul. 46 Revised or Bul. 107).

(2) Proposed volumetric method for potash in soils.

Reagents.

Nitric acid.—50.0 cc nitric acid 1.40 sp. gr. in 1,000 cc water.

Sodium nitrate wash.—10.0 grams sodium nitrate per 1,000 cc water.

Phosphomolybdic acid solution.—100 grams phosphomolybdic acid (Kahlbaum's preferred) in 750 cc water and 250 cc nitric acid (1.40 sp. gr.). This solution must be freshly prepared—not over three or four days old before using. If properly prepared, the evaporated residue from a portion of this solution is never white and readily redissolves in the dilute nitric acid solution in the cold.

Standard solutions.—Standard caustic potash and nitric acid prepared for volumetric phosphoric acid; 1 cc potassium hydroxid is equal to 1.625 mg K_2O .

Determination.

Place a fresh aliquot, representing 1 gram of soil of solution A under soil analysis (Bul. 107 Revised, p. 14), in a tall 200 cc beaker, add 20 cc phosphomolybdic solution, and slowly evaporate to complete dryness on a steam bath. If preferred, use solution without removal of silica, evaporating the acid soil solution directly with phosphomolybdic acid. (It requires approximately 22 mg of phosphomolybdic acid in order to have an excess for each milligram of potassium monoxid present.)

Add 30 cc of nitric acid wash to the dried residue, stir thoroughly in the cold with a grinding motion, using a policeman, allow to settle a moment, and decant supernatant liquid at once through a gooch crucible packed with moist filter-paper pulp approximately one-sixteenth inch in thickness. Wash twice by decantation with sodium nitrate wash, transfer precipitate to gooch, and wash with sodium nitrate wash until acid-free. Transfer gooch to casserole, run in excess of standard alkali solution, and add phenolphthalein. Heat to boiling and titrate excess of alkali with standard acid.

Note.—Some samples of asbestos used seemed to hold or "fix" some of the excess acid, making it very hard to wash the gooch filter acid-free. Hence it is suggested to use a paper-pulp filter.

It is suggested that the evaporated residue in the beaker be transferred to a centrifuge tube instead of a gooch, using nitric acid wash, whirl until well settled, decant, and wash with sodium nitrate wash, using the centrifuge, until acid-free. Dissolve in standard alkali and titrate as above.

WORK ON MIXED FERTILIZERS.

Mix thoroughly before sampling.

(1) In sample No. 2 determine water-soluble potash (K_2O) by the official method (U. S. Dept. Agr., Bureau of Chemistry Bul. 46 Revised, p. 21, or Bul. 107, p. 11).

(2) Use proposed volumetric method for potash in fertilizers. Transfer 10 cc of solution No. 2 (Bul. 46 Revised, pp. 21-22, or Bul. 107, pp. 11-12) to a platinum dish, and add 0.25 cc of sulphuric acid (1 to 1). Evaporate to dryness and ignite to whiteness. Dissolve residue in hot water plus a few drops of hydrochloric acid and transfer to a tall 200 cc beaker; add 30 cc of phosphomolybdic acid solution and proceed as under proposed volumetric method for potash (K_2O) in soils. (If excess of phosphomolybdic acid has been used, the dried residue has a reddish hue. If excess has not been added, the residue is bright yellow. The residue should not appear white.)

In each case run blanks to ascertain corrections to be made for impurities. It is also advisable to treat the potassium-platinic-chlorid residue in the gooch crucible in order to ascertain if it is all soluble in water. Then reweigh the crucible after thoroughly drying.

Note.—The caustic potash solution must be free from carbonate. (See Sutton, Volumetric Analysis, 9th edition, revised, page 48, for the barium hydrate method, also page 301 for the alcoholic method for making potassium hydroxide free from carbonates.)

A. L. KNISELY, *Referee.*

B. B. ROSS, *Associate Referee.*

Ten sets of samples were sent out and three reports have been received.

TABLE V.—*Comparison of official and proposed volumetric method for the determination of potash in soil and in mixed fertilizers.*

Analyst.	Soil No. 1.		Fertilizer No. 2.		
	Official.	Volumetric.	Official water-soluble.	Volumetric.	Official—burning off with $(NH_4)_2SO_4$ instead of H_2SO_4 .
M. G. Donk, Bureau of Chemistry, Washington, D. C.....	<i>Per cent.</i> 0.35 .37 .39 .39 42 .43	<i>Per cent.</i> 0.44 .38 .40 .46 42 .43	<i>Per cent.</i> 10.59 10.65 10.62	<i>Per cent.</i>	<i>Per cent.</i> 10.55 10.53 10.66
A. L. Knisely and C. E. Bradley, Oregon station.....	.42 .42 .42	.39 .41 .38	10.65 10.71	10.82 10.65	
S. E. Asbury, Texas station.....	<i>a</i> .50 <i>a</i> .55	.40 .52	<i>a</i> 11.11 <i>b</i> 11.14	11.62 11.89 11.92	
E. Carlyle, Texas station.....		.32 .32 <i>c</i> .38			

a Direct weight.

b Washed out.

c By volumetric method formerly used.

d No correction for reagent used.

e Washed by centrifuge.

COMMENTS BY ANALYSTS.

M. G. Donk: Could obtain no satisfactory results on sample No. 2 with the volumetric method, presumably due to difficulty in washing yellow precipitate.

G. S. Fraps: Sample of phosphomolybdic acid from Merck left considerable white residue, probably molybdic acid, when diluted in nitric acid. This probably accounts for volumetric results being higher than the official method.

C. E. Bradley: Best results were always obtained when using small amounts of potash solution. An aliquot part of a solution of fertilizer containing not more than 0.02 gram K_2O gave the best results. If, after evaporating to dryness and taking up in dilute nitric acid, the insoluble residue is not bright yellow, then the conditions are not normal. The washed precipitate should be bright yellow.

COMMENTS BY REFEREE.

It was recommended this year to use a stronger nitric acid wash solution than was formerly used. This was on account of the difficulty experienced in washing the yellow precipitate free from impurities. It has since been ascertained that this difficulty was due to poorly prepared or old phosphomolybdic acid solution. The phosphomolybdic acid solution, when prepared from good, fresh chemicals, should give no blank, and when used in volumetric potash work dissolves and washes out very readily from the evaporated residue. It has recently been found that the nitric acid wash, instead of being stronger than was formerly used, should, if anything, be weaker. In some cases when determining potash in material containing considerable calcium and magnesium and moderate quantities of iron, such as some soil solutions, it is not necessary to use a nitric acid wash solution, but only to dissolve the material at once in the sodium nitrate wash solution.

Official soil solution evaporated to dryness with phosphomolybdic acid and taken up in nitric acid wash gave 0.45 per cent K_2O ; when taken up directly in sodium nitrate wash the same result was obtained.

I think the merit of the volumetric method for potash depends entirely upon the phosphomolybdic acid solution and the care with which the evaporation is conducted. The difficulties thus far experienced with blanks and in washing the evaporated residues have been largely due to the presence of white insoluble molybdic acid, which at times separates during the process of evaporation and is difficult to remove from the yellow residue of potassium phosphomolybdate.

RECOMMENDATION.

The referee recommends a continuation of the study of the volumetric method for use in both soil and fertilizer analysis; also that the recommendation of Mr. Cushman concerning a study of what really constitutes available potash in soils, fertilizers, and ground-mineral products be continued, so that if possible we may have a definition of available potash.

REPORT ON INSECTICIDES.

By R. J. DAVIDSON, *Referee*.

The suggestions made in the recommendations of last year were followed as closely as possible, and after consultation with the associate referee the following work was outlined:

- (1) Work on London purple to test the Davidson modification of Haywood's method in comparison with the method as originally outlined.
- (2) Tests to determine which of the two methods of analysis of soda lye give the more correct results.
- (3) Testing the formaldehyde method as modified by Haywood and Smith, in comparison with the method as it originally stood.
- (4) Testing the Avery method of determining sulphur in sulphur dips.
- (5) Testing the methods of examining lead arsenate as published in last year's proceedings.

Requests for cooperation were sent to thirty chemists. Of these, but two signified their willingness to undertake the work. One report contained results obtained by two analysts in the same laboratory. The associate referee was prevented from doing any work on account of a serious injury he received as the result of an accident in his laboratory. The report therefore contains the results of five analysts.

LONDON PURPLE.

The work on London purple consisted in testing the modified Haywood method (for the removal of part of the coloring matter) in comparison with the method as originally outlined. The modification was proposed in 1903 and was given a partial trial in 1904, but no definite results were obtained. A statement was made that it greatly facilitated end point readings. The benefits of this modification are twofold: Time is saved and, by removing a part of the coloring matter, the end point reading in the titration is made less difficult. If, therefore, the modification does not interfere with its accuracy, it will be of great advantage.

In presenting the work done it has been deemed advisable to give the results for arsenious and arsenic oxids in separate tables, as the work on these two determinations has not been equally satisfactory. The subject will therefore be discussed under two heads.

TOTAL ARSENIUS OXID.

Method I is a provisional method, and may be found in Circular No. 10, Revised, Bureau of Chemistry.

Method II is the proposed modified method, and differs from Method I in that the process of filtering and washing to a definite volume after dissolving the material is eliminated, and in addition a part of the coloring matter is precipitated. The method is as follows:

Place 2 grams of London purple in a beaker and dissolve in about 80 cc of water and 20 cc of concentrated hydrochloric acid at a temperature of 60° to 70° C.; cool, and add sodium carbonate in slight excess, transfer to a 250-cc flask, bring to mark, shake, and filter through a dry filter. Acidify 50 cc of the filtrate with hydrochloric acid and make alkaline with sodium bicarbonate. Titrate the amount of arsenious oxid present with standard iodine solution.

TABLE I.—*Total arsenious oxid.*

Analyst.	Method I.	Method II
	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	16.78 16.94 16.86 16.83	16.43 16.50 16.47 16.41
H. R. Watkins, Pullman, Wash.....	16.83 16.83 16.54	16.67 16.54 16.33
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.....	16.54 16.72 16.72	16.25 16.33 16.22
R. R. Henley, Blacksburg, Va.....	16.70 16.75	16.31 16.54
R. J. Davidson, Blacksburg, Va.....	16.70 16.75	16.35 16.54
Average.....	16.75	16.42

Discussion.

The results upon total arsenious oxid agree very well. There is a difference of only 0.33 per cent between the averages by the two methods, showing that

in case of the total arsenious oxid, the shorter method gives practically the same results.

TOTAL ARSENIC OXID.

Methods I and II are the same as were used in 1904 and 1905, and may be found in Circular No. 10, Revised, of the Bureau of Chemistry. Method II is a modification of Method I, the difference being that before proceeding with the reduction of the solution a part of the coloring matter is removed by precipitation with sodium carbonate. Method III is a still further modification of Method I, as will be seen by reference to Method II under total arsenious oxid.

Method III reads as follows:

Acidify 50 cc of the solution prepared under determination of total arsenious oxid, Method II, with concentrated hydrochloric acid, heat to 80° C., add 50 cc more concentrated hydrochloric acid and 3 grams of potassium iodid, and proceed as described in Method I for total arsenic oxid.

The only difference between Methods II and III is the elimination of the filtering and washing to definite volume of the hydrochloric acid solution, which saves considerable time

TABLE II.—*Total arsenic oxid.*

Analyst.	Method I.	Method II.	Method III.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	12.43 12.12 12.27	11.04 11.15 11.10	9.73 10.09 9.86
H. R. Watkins, Pullman, Wash.....	12.91 13.06 12.98	9.93 10.08 10.00	9.55 9.45 9.50
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.....	13.15 13.25 12.95	13.05 13.25 12.95	12.98 12.98 12.90 12.94
R. R. Henley, Blacksburg, Va.....	12.67 12.98 12.52	10.14 10.34	9.86 10.43
R. J. Davidson, Blacksburg, Va.....	12.66 12.95 12.51	10.35	10.75 9.96

Discussion.

The results upon total arsenic oxid are very unsatisfactory. There is a fairly good agreement of the results of four analysts under each method, but the results of the different methods do not agree, there being a difference of about 2 per cent between Method I and Methods II and III. One set of results shows an excellent agreement by all three methods. All of the results agree very well by Method I, but by Methods II and III one set of results is about 2 per cent higher than the other four.

It is very difficult, if not impossible, to explain why four sets of results should agree so well among themselves by Methods II and III, yet differ so widely from the other set. If there had been agreement between all the analysts, then the difference between Method I and Methods II and III might be attributed to one of two things: an error in Method I due to the difficulty of reading accurately the end point in the titration when so much coloring matter is present, or to the loss of arsenic occurring in removing the coloring matter. It was suggested that the difference was partly due to not carrying out properly the details of Method III, as it was thought that the directions for Method III were not explicit enough as to heating the solution before adding

hydrochloric acid and using only 25 cc instead of 50 cc. This can not be the explanation, for in my own work the solution was heated to 80° C. and 50 cc concentrated hydrochloric acid tried as well as 25 cc, giving practically the same results. It is regretted that no comments are made by the analysts on this point, and also that the referee did not have time to investigate it further. Method II was used in 1903 and 1904, but no mention was made of any difficulty occurring; in fact, the referee stated in his discussion that "the modification suggested for the removal of a part of the coloring matter works very well, greatly facilitating the end point reading." In applying this method to the samples of 1903, I did not notice such a difference in results. This part of the work on London purple must be repeated next year, and it is to be hoped that the cause of the discrepancy will be discovered.

SODA LYE.

The methods used for the examination of soda lye were the same as have been used for several years, and may be found in Circular No. 10, Revised, Bureau of Chemistry.

In Method I the total alkalinity is obtained by titrating an aliquot of the dilute solution with half-normal acid, using methyl orange as an indicator. Another aliquot is taken, and the carbon dioxid of the carbonate is precipitated by a dilute solution of barium chlorid and then made to a definite volume. The precipitate is allowed to settle, and an aliquot of this is taken and titrated with half-normal acid, using phenolphthalein as an indicator. In Method II an aliquot is titrated with acid potassium sulphate, using phenolphthalein and methyl orange as indicators.

TABLE III.—*Soda lye.*

Analyst.	Method I.		Method II.	
	Sodium hydroxid.	Sodium carbonate.	Sodium hydroxid.	Sodium carbonate.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	88.91	5.29	91.53	5.44
H. R. Watkins, Pullman, Wash.....	89.12	5.00	90.80	5.13
J. K. Haywood and C. C. McDonnell, Washington, D. C. {	91.82	3.30	91.25 91.15	4.18 4.18
	91.71	2.74		
	91.43	2.75		
R. R. Henley, Blacksburg, Va.....	91.46	3.56	91.59	4.67
	92.09	3.93		
R. J. Davidson, Blacksburg, Va.....	91.73	3.56	91.04	4.70
	92.04	3.40		

DISCUSSION.

The results are not so good as they have been in previous years. Two sets of results are slightly lower in sodium hydroxid and higher in sodium carbonate than the others. The results for sodium hydroxid by the two methods agree very well. The sodium carbonate by Method II is higher. This higher result may be due to the difficulty in reading the end point with methyl orange. In Method II the titration is tedious, and for this reason, together with the difficulty in reading the end point in the titration, the referee thinks it should be dropped from the methods. In Method I, when titrating with phenolphthalein to determine the sodium hydroxid after precipitating with barium chlorid, it is not necessary to filter off the precipitate, as has been pointed out before, since the precipitate does not interfere with the titration. The results obtained when the precipitate is permitted to remain in the solution are the same as when it has been removed. Better results would probably be obtained in this work if a fifth normal solution of hydrochloric acid were used instead of half normal. A

difference of 0.1 cc of half-normal solution makes a difference of 0.6 per cent in the result.

FORMALDEHYDE.

The methods used for the analysis of formaldehyde were the modified hydrogen peroxid method proposed by Mr. Haywood and the cyanid method.

TABLE IV.—*Formaldehyde.*

Analyst.	Hydrogen peroxid method.	Potassium cyanid method.
	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	36.24	35.24
	36.42	35.24
	36.33	35.24
H. R. Watkins, Pullman, Wash.....	36.24	34.50
	36.15	34.79
	36.20	34.65
J. K. Haywood and C. C. McDonnell, Bureau of Chemistry.....	36.00	}
	36.18	
	36.15	
R. R. Henley, Blacksburg, Va.....	36.30	35.22
	36.30	35.22
R. J. Davidson, Blacksburg, Va.....	36.30	35.20
	36.30	35.21

COMMENTS OF ANALYSTS.

J. K. Haywood.—The results on formaldehyde are no doubt lower than they would have been had the determination been made when the sample was received, as some insoluble polymerized formaldehyde settled on the bottom of the bottle during the time intervening between its receipt and its analysis. The acidity of the hydrogen peroxid used was determined and correction made therefor.

DISCUSSION.

The results as a whole are exceedingly good. Method II can only be used with a solution containing a small quantity of formaldehyde; therefore it seems advisable to state the strength of the solution more definitely—for example, about 1 per cent or 2 per cent solution, instead of specifying “a small quantity.” This point is referred to the next referee for investigation, and it is recommended that the hydrogen peroxid method be made official.

SULPHUR DIPS.

The method for sulphur dips was the same as that tested by the referee in 1905, and is given in Circular No. 10, Revised, Bureau of Chemistry.

TABLE V.—*Sulphur dips.*

Analyst.	Month of analysis.	Weight of sulphur.	Sulphur by weight.
		<i>Gram per cc.</i>	<i>Per cent.</i>
	1907.	0.0484	4.61
R. W. Thatcher, Pullman, Wash.....	June.....	.0484	4.61
		.0484	4.61
H. R. Watkins, Pullman, Wash.....	June.....	.0456	4.35
		.0455	4.34
		.0456	4.36
J. K. Haywood and C. C. McDonnell, Washington, D. C.....	June.....	.0465	}
		.0466	
		.0466	
R. R. Henley, Blacksburg, Va.....	June.....	.0416	}
		.0427	
R. J. Davidson, Blacksburg, Va.....	June.....	.0431	}
		.0435	

DISCUSSION.

The results are all good, the difference between the lowest and the highest being about 0.5 per cent. The method is easy of manipulation and very satisfactory. I would recommend that it be adopted as an official method.

LEAD ARSENATE.

The methods used for lead arsenate were those proposed by Mr. Haywood at the last meeting of the association, and are found in the Proceedings of 1906.^a

The material used for analysis was purchased from the same person from whom the other samples used in this work were obtained, and when received was in the form of a thick paste. It was dried and ground before being sent out.

TABLE VI.—*Lead arsenate.*

Analyst.	Moisture.	Total arsenic oxid.	Total lead oxid.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
R. W. Thatcher, Pullman, Wash.....	1.37	25.57	65.10
	1.40	25.72	64.83
	1.38	25.64	64.95
H. R. Watkins, Pullman, Wash.....	1.50	25.31	64.49
	1.50		64.34
		24.74	66.70
J. K. Haywood and C. C. McDonnell, Washington, D. C.....	1.18	24.57	66.73
		24.40	66.01
		24.57	66.88
R. R. Henley, Blacksburg, Va.....	1.00	24.41	65.40
		24.60	65.40
R. J. Davidson, Blacksburg, Va.....	1.00	24.44	65.72
		24.66	65.44

DISCUSSION.

The results are not as good as they should be. On total lead oxid there is a difference of over 2 per cent between the lowest and highest, and on the total arsenic oxid the difference is over 1 per cent. I would recommend that these methods be further tried by the next referee.

RECOMMENDATIONS.

It is recommended—

(1) That the Haywood-Davidson provisional method for determining total arsenic oxid in London purple be given a further trial, and that this be compared with the original provisional method.

(2) That a further study of the amount of hydrochloric acid used in the reduction of London purple be made in order to determine if 25 cc can be used instead of 50 cc.

(3) That the provisional precipitation method for the analyses of soda lye be referred to the referee with a view to its adoption as official.

(4) That the provisional potassium acid sulphate method for the analysis of soda lye be dropped from the methods of analysis.

(5) That the provisional hydrogen peroxid method for the determination of formaldehyde be referred to the referee with a view to its adoption as official.

^a U. S. Dept. Agr., Bureau of Chemistry, Bul. 105, page 165.

(6) That the cyanid method for the determination of formaldehyde be further studied in order to determine more definitely what strength of formaldehyde may be estimated by this method.

(7) That the provisional hydrogen peroxid method for the determination of sulphur in sulphur dips be referred to the referee with a view to its adoption as official.

(8) That the methods proposed by Mr. Haywood for the analyses of lead arsenate be made provisional and further studied with a view to their adoption by the association.

REPORT OF COMMITTEE A ON RECOMMENDATIONS OF REFEREES.

By R. J. DAVIDSON, *Chairman*.

(Phosphoric acid, potash, nitrogen, soils, ash, and insecticides.)

(1) NITROGEN.

It is recommended—

(1) That "20 cc of sulphuric acid," line 3, under (3) Determination, page 6, Bulletin 107, on Methods of Analysis, be changed to read "from 20 to 30 cc of sulphuric acid."

Adopted.

(2) That in line 4, same reference, the following phrase be inserted after the word "acid:": "from 0.1 to 0.3 gram of crystallized copper sulphate may also be used in addition to the mercury or in lieu of it."

Referred to the referee for 1908 with a recommendation for adoption.

(3) That following the sixth line from the bottom of page 6, same reference, after the words "standard acid," this sentence be added: "During at least the first ten minutes of distillation the lower end of the condenser tube should dip beneath the surface of the standard acid."

The motion to adopt this recommendation was lost.

(4) That on page 7 of Bulletin 107, under "(b) Gunning method, (3) Determination," line 4, after the words "sulphuric acid" the following phrase be inserted: "from 0.1 to 0.3 gram of crystallized copper sulphate may also be added."

Referred to the referee for final action as to adoption in 1908.

(5) That on page 7 of Bulletin 107, after insert recommended under (4), add the following:

Approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury may also be added, before the addition of the potassium sulphate, but if mercury be used potassium sulphid must be employed, as in the Kjeldahl method, in the distillation.

This recommendation was referred to the committee on the revision of methods to be inserted as a special method instead of being introduced as the referee recommended.

(2) POTASH.

It is recommended—

(1) That since the volumetric method for potash, especially in soils, saves much time, the study of this method for use in soil and fertilizer analysis be continued; and further that the recommendation made by Mr. Cushman in 1906, to the effect that the referee "turn his attention to a definition of available potash," be made the basis for work the coming year.

Adopted.

(3) PHOSPHORIC ACID.

It is recommended—

(1) That Thomas or basic slag be prepared for analysis as are other fertilizers or fertilizing materials under the methods of this association.

Adopted provisionally pending further work.

(2) That the solution for analysis be made according to (a₇), page 2, Bulletin 107, under "(2) Total phosphoric acid." or in strong hydrochloric acid alone. In the latter case, after the portion for analysis is measured out, add nitric acid and heat for a few minutes. Then determine total phosphoric acid according to the official methods.

Adopted provisionally pending further work.

(3) That the fineness of the material be determined according to the plan followed with bone meal and the commercial value estimated on the basis of total phosphoric acid and fineness of product.

Adopted provisionally pending further work.

(4) That the referee further study the subject of basic slag with a view to devising some method for the determination of the available phosphoric acid which it contains.

This recommendation was made by Committee A and was adopted.

(4) INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the peroxid method remain provisional [not adopted as official as recommended by the referee].

Adopted.

(2) That the combustion method be adopted as provisional.

Adopted.

(3) That the method for ash, as given in Bulletin 46, page 77, be readopted as official.

Adopted.

(4) That the development of methods for determining iron and aluminum in ash be referred to the referee for 1908.

Adopted.

(5) SOILS.

It is recommended—

(1) That the modified J. L. Smith method for total potassium be further tested. (Circular 32, p. 4.)

Adopted.

(2) That the sodium peroxid fusion method for total phosphorus be adopted as a provisional method of this association and be further tested. (Bul. 99, p. 111; Bul. 105, p. 145.)

Adopted.

(3) That the determination of volatile matter (page 14, Bulletin 107, section 4) be replaced by the "Determination of Total Organic Carbon" (J. Amer. Chem. Soc., 1904, 26: 1640).

In lieu of this it was recommended by Committee A that the method suggested for the determination of total organic carbon be referred to the committee on revision of methods for insertion as an additional determination, the original method to be also retained.

The recommendation of Committee A was adopted.

(6) INSECTICIDES.

It is recommended—

(1) That the Haywood-Davidson provisional method for determining total arsenic oxid in London purple be given a further trial, and that this be compared with the original provisional method. (Bul. 107, pp. 28, 29; also Circular 10.)

Adopted.

(2) That a further study of the amount of hydrochloric acid used in the reduction of London purple be made in order to determine whether 25 cc can be used instead of 50 cc.

Adopted.

(3) That the provisional precipitation method for the analysis of soda lye be made official. (Bul. 107, p. 31; also Circular 10.)

Referred to the referee for 1908 for final recommendation.

(4) That the provisional potassium acid sulphate method for the analysis of soda lye be dropped from the methods of the association.

Adopted.

(5) That the provisional hydrogen peroxid method for the determination of formaldehyde be made official. (Bul. 107, p. 33.)

Referred to the referee for 1908 for final recommendation.

(6) That the cyanid method for the determination of formaldehyde be further studied in order to determine more definitely what strength of formaldehyde may be estimated by this method. (Bul. 107, p. 33.)

Adopted.

(7) That the provisional hydrogen peroxid method for the determination of sulphur in sulphur dips be made official. (Bul. 107, p. 34.)

Referred to the referee for 1908 for final recommendation.

(8) That the methods proposed by J. K. Haywood for the analysis of lead arsenate (Proceedings 1906, Bul. 105, p. 165) be made provisional, and further studied with a view to their adoption by the association.

Adopted.

The association voted to hold the convention of 1908 in Washington and the meeting adjourned.

OBITUARY.

GEORGE CHAPMAN CALDWELL.
(1834-1907.)

WILBUR OLIN ATWATER.
(1844-1907.)

During the month of September, 1907, the death of two earnest and gifted workers in the field of scientific agriculture was reported, both chemists who worked with such enthusiasm and insight as to inspire all with whom they came in contact.

Dr. George Chapman Caldwell was for many years one of the leading writers and lecturers on agricultural experimentation, and the book "Agricultural Chemical Analysis," which he published in 1869, was the first of its kind and for a long time the English standard for agricultural science. He chose for his teachers both here and abroad the most famous scholars, and, as a consequence, gained a deep and broad knowledge of his subject. After having taught in several other colleges Doctor Caldwell was called to Cornell in 1868, being the first professor appointed. There he held the chair of chemistry for thirty-five years, during which time he served not only as teacher, but also as director of the experiment station of the university. In 1879, when the Cornell University Agricultural Experiment Station was organized, he was put at its head; and in 1887, when the station was reorganized, Doctor Caldwell was appointed chemist and retained this office until his retirement, twenty-five years later. While holding this directorship he published many valuable papers on the results of his work on fertilizers, cattle foods, sugar-beet culture, and methods of agricultural analysis.

He sought to inspire his students with the idea that thoroughness and accuracy were the fundamental principles of any work of lasting value. To his mind the possibilities for useful investigation in the field of agricultural science were unlimited, and he filled all who came in contact with him with equal enthusiasm, and in this way his ideals of work are perpetuated in his students and fellow-workers.

Dr. Wilbur Olin Atwater was an untiring laborer for the application of scientific methods to agriculture, and it was largely due to him that experiment stations were established in the United States.

When the first State experiment station was opened in Connecticut Doctor Atwater was appointed director. As other States realized the usefulness of this station and established similar ones, Doctor Atwater interested himself in them all, so that when the time came to demand national support he was fully equipped to represent the cause. In 1887, after the passage of the Hatch Act, the Storrs Agricultural Experiment Station was established, and Doctor Atwater was appointed its director. Much excellent work was done here in the twelve years during which he retained this directorship. Among the most interesting publications issued by the station was the continuation of Doctor

Atwater's studies on the absorption of atmospheric nitrogen by plants, studies which he had instituted as professor of chemistry at Wesleyan University.

When the work of the State experiment stations became so important that it was decided to establish an Office of Experiment Stations in the United States Department of Agriculture Doctor Atwater was summoned to take charge of it in addition to his other work. It was in this position that his wonderful executive ability had full play, for he aimed to make this national office a "clearing house and exchange for the State experiment stations." But with all his practical understanding Doctor Atwater was primarily a scientific investigator. His high ideals in this regard are expressed by his own statement: "The future usefulness of the stations will depend upon what they discover of permanent value, and this must come largely from the most abstract and profound research; to forget this will be fatal."

In 1889 he instituted the publication of a series of monographs on special subjects, under the name of Farmers' Bulletin, and these proved so popular that Congress appropriated a printing fund for them and arranged for their distribution on a large scale. The publication of the Experiment Station Record also was inaugurated by Doctor Atwater and proved of great value to agricultural scientists.

As the usefulness of the central office increased the work became too heavy for its director, who did not wish to relinquish entirely his work of teacher and investigator, and he therefore resigned, although he retained close relations with the office until the end of his active career.

Parallel with his studies in agricultural chemistry, Professor Atwater began early in his career to carry on investigations in physiological chemistry with special reference to human nutrition. In the early eighties, after a special study of problems and methods, he made the subject of human nutrition an important part of the work of the Connecticut Storrs Agricultural Experiment Station. Among the important results of this work may be mentioned the elaboration of experimental methods, the improvement of the bomb calorimeter, and the development of the respiration calorimeter in such form that the income and outgo of both matter and energy can be measured.

The accuracy of his scientific methods, the scope of his investigations, and his splendid understanding of practical conditions make his work valuable for all time. This, and the personality of Doctor Atwater, won the confidence of others, so that it was possible for him to carry out his ideas in successful practice. It was the rare combination in Doctor Atwater of a well-trained mind, splendid ability to organize and manage, and high scientific ideals that enabled him to be of such great service to humanity.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1908.

President.

Mr. HARRY SNYDER, St. Anthony Park, Minn.

Vice-President.

Mr. W. D. BIGELOW, Washington, D. C.

Secretary.

Mr. H. W. WILEY, Washington, D. C.

Additional members of the Executive Committee.

Mr. B. B. ROSS, Auburn, Ala.

Mr. G. S. FRAPS, College Station, Tex.

Referees.

Phosphoric acid: J. M. McCandless, Atlanta, Ga.

Nitrogen:

Determination of nitrogen: C. L. Penny, State College, Pa.

Separation of nitrogenous bodies: L. L. Van Slyke, Geneva, N. Y. (milk and cheese proteids).

Potash: B. B. Ross, Auburn, Ala.

Soils: S. D. Averitt, Lexington, Ky.

Dairy products: J. M. Bartlett, Orono, Me.

Foods and feeding stuffs: J. P. Street, New Haven, Conn.

Food adulteration: H. E. Barnard, Indianapolis, Ind.

Sugar: A. H. Bryan, Washington, D. C.

Tannin: F. P. Veitch, Washington, D. C.

Insecticides: C. C. McDonnell, Washington, D. C.

Inorganic plant constituents: H. D. Haskins, Amherst, Mass.

Medicinal plants and drugs: L. F. Kebler, Washington, D. C.

Associate referees.

Phosphoric acid: W. F. Hand, Agricultural College, Miss.

Nitrogen:

Determination of nitrogen: G. W. Cavanaugh, Ithaca, N. Y.

Separation of nitrogenous bodies:

Meat proteids: P. F. Trowbridge, Columbia, Mo.

Vegetable proteids: E. B. Hart, Madison, Wis.

Potash: E. L. Baker, Geneva, N. Y.

Soils: J. G. Lipman, New Brunswick, N. J.

Dairy products: L. G. Michael, Ames, Iowa.

Foods and feeding stuffs: F. W. Morse, Durham, N. H.

Food adulterations:

Colors: H. M. Loomis, Galveston, Tex.

Saccharine products: Arthur Given, Washington, D. C.

Fruit products: C. B. Cochran, West Chester, Pa.

Wine: Julius Horvet, St. Paul, Minn.

Beer: H. E. Barnard, Indianapolis, Ind.

Food adulterations—Continued.

- Distilled liquors: L. M. Tolman, Washington, D. C.
 Vinegar: Chas. H. Hickey, Boston, Mass.
 Flavoring extracts: E. M. Chace, Washington, D. C.
 Spices: A. L. Winton, Chicago, Ill.
 Baking powder and baking chemicals: E. L. Redfern, Lincoln, Nebr.
 Meat and fish: F. C. Weber, Washington, D. C.
 Fats and oils: H. E. Bishop, Indianapolis, Ind.
 Dairy products: Herman Lythgoe, Boston, Mass.
 Cereal products: E. F. Ladd, Fargo, N. Dak.
 Vegetables: W. L. Dubois, Buffalo, N. Y.
 Condiments other than spices: E. M. Bailey, New Haven, Conn.
 Cocoa and cocoa products: A. G. Woodman, Boston, Mass.
 Tea and coffee: A. G. Woodman, Boston, Mass.
 Preservatives: W. D. Bigelow, Washington, D. C.
 Determination of water in foods: E. W. Brown, Washington, D. C.

Sugar:

- Molasses methods: H. P. Agee, Audubon Park, La.
 Chemical methods: Fritz Zerban, Audubon Park, La.

Tannin: M. S. McDowell, State College, Pa.

Insecticides: Howard R. Watkins, Pullman, Wash.

Inorganic plant constituents: F. W. Robison, Lansing, Mich.

SPECIAL COMMITTEES.

Food Standards.

- Mr. William Frear, State College, Pa., chairman.
 Mr. H. W. Wiley, Washington, D. C.
 Mr. H. A. Weber, Columbus, Ohio.
 Mr. M. A. Scovell, Lexington, Ky.
 Mr. E. H. Jenkins, New Haven, Conn.

Fertilizer Legislation.

- Mr. H. W. Wiley, Washington, D. C., chairman.
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Revision of Methods.

- Mr. J. K. Haywood, Washington, D. C., chairman.
 Mr. F. P. Veitch, Washington, D. C.
 Mr. L. M. Tolman, Washington, D. C.
 Mr. J. P. Street, New Haven, Conn.
 Mr. A. L. Winton, Chicago, Ill.
 Mr. J. H. Pettit, Urbana, Ill.
 Mr. F. W. Woll, Madison, Wis.

Committee to present the question of the unification of terms to the International Congress of Applied Chemistry.

Mr. R. J. Davidson, Blacksburg, Va., chairman.

Mr. C. G. Hopkins, Urbana, Ill.

Mr. W. D. Bigelow, Washington, D. C.

Mr. G. S. Fraps, College Station, Tex.

Mr. B. W. Kilgore, Raleigh, N. C.

Mr. H. J. Wheeler, Kingston, R. I.

Mr. J. T. Willard, Manhattan, Kans.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statement of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officio and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, from among the members of the association, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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